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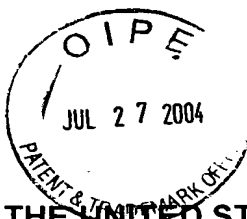
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1733 JFW

Patent  
Attorney's Docket No. 032887-007

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of	)	<b>MAIL STOP AMENDMENT</b>
Tadashi KITAMURA et al.	)	
Application No.: 10/069,048	)	Group Art Unit: 1733
Filed: February 21, 2002	)	Examiner: Steven D. MAKI
For: SEALANT COMPOSITION FOR	)	Confirmation No.: 9296
PLASTIC LIQUID CRYSTAL	)	
DISPLAY CELL	)	

**SUBMISSION OF VERIFIED TRANSLATION OF PRIORITY DOCUMENT**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:


In accordance with the statement set forth on page 14 of the Amendment filed on June 28, 2004, provided herewith is a verified English translation of the Japanese priority application, Patent Application No. 2000-185871, filed on June 21, 2000. As may be seen therefrom, the claims under consideration, such as claim 3 relating to a sealant composition comprising a two-component epoxy resin composition, can find support in the priority application. For instance, claim 5 of the priority application also relates to a sealant composition which is a two component epoxy resin composition. In the present application, the percentage of the liquid epoxy has been rounded off and the ionic conductivity uses different units. In addition, the types of polyfunctional epoxy resins described in the passage starting on page 25 of the present application follows the description starting on page 43 of the translation. The description of the tetrafunctional mercapto compounds and derivatives as curing agents starts on page 53. Rubbery polymer fine particles are disclosed in the

passage starting at the top of page 92 of the translation with the description including a softening temperature of 0° or less and a particle size of 0.01 to 5 microns.

By conducting a review of the provided translation, it is apparent that the claims under consideration are entitled to the filing date of the priority application which is prior to publication date of JP 2000-347203. Accordingly, for the reasons provided in the remarks of the previously filed Amendment, reconsideration and allowance of the present application are respectfully requested.

Respectfully submitted,

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Date: July 27, 2004



#### DECLARATION

I, Emiko Amano declare that:

1. I reside at c/o Saikyo Patent Office, Shikishima Building 6th Floor, 2-6, Bingomachi 3-chome, Chuo-ku, Osaka, Japan.

2. I understand and read both the Japanese and the English languages.

3. The attached is a full true and faithful English translation made by me of the priority document of the Japanese Patent Application No. 2000-185871 filed on June 21, 2000.

4. I declare further that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-identified application or any patent issuing thereon.

Date: July 27, 2004 Name: Emiko Amano  
Emiko Amano



**PATENT OFFICE  
JAPANESE GOVERNMENT**

This is to certify that the annexed is a true copy  
of the following application as filed with this Office.

Date of Application : June 21, 2000

Application Number : Japanese Patent Application  
No. 2000-185871

Applicant : MITSUI CHEMICALS INC.

July 27, 2001

Commissioner, Kozo OIKAWA  
Patent Office

(seal)

Certificate Issue Number 2001-3065575



[DOCUMENT] APPLICATION FOR PATENT

[REFERENCE] 41000142

[FILING DATE] June 21, 2000

[ADDRESSEE] To Commissioner of the Patent Office

[IPC] G02F 1/1339  
C08G 59/56  
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[IDENTIFICATION OF OFFICIAL FEE]  
[Prepaid No.] 005278  
[Official Fee] 21,000

[LIST OF THE ATTACHED DOCUMENTS]  
[Item] Specification 1  
[Item] Abstract 1

SPECIFICATION

[DOCUMENT] SPECIFICATION

[TITLE OF THE INVENTION] SEALANT COMPOSITION FOR PLASTIC  
LIQUID CRYSTAL CELL

[CLAIMS]

[Claim 1] A sealant composition for a plastic liquid  
crystal cell, comprising:

an epoxy resin composition, wherein

(I) an aqueous solution obtained by mixing the  
composition and 10 times by weight of pure water at from  
40 to 80°C has an ionic conductivity of 10  $\mu\text{S}/\text{cm}$  or less,  
and

a cured product of the composition has

(II) a water vapor permeability at 60°C passing a  
cured film of the composition having a thickness of 100  $\mu\text{m}$   
of less than 200  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ ,

(III) a heat deformation temperature ( $T_g$ ) of a cured  
product of the composition in a range of from 0 to 90°C,  
and

(IV) a storage modulus under room temperature of a  
cured product of the composition in a range of from  $0.5 \times 10^4$   
to  $1 \times 10^6$  Pa.

[Claim 2] The sealant composition for a plastic liquid

crystal cell of claim 1, wherein the epoxy resin composition has

(V) an E type viscosity at from 50 to 100°C after coating to a thickness of 50  $\mu\text{m}$  and being subjected to a heat treatment at from 50 to 85°C for 20 minutes of from 5 to 5,000 Pa·s.

[Claim 3] A sealant composition for a plastic liquid crystal cell of claim 1 and 2, wherein the epoxy resin composition is a two-component epoxy resin composition.

[Claim 4] A sealant composition for a plastic liquid crystal cell of claim 1 and 2, wherein the epoxy resin composition is a one-component epoxy resin composition.

[Claim 5] A sealant composition for a plastic liquid crystal cell, wherein the epoxy resin composition is a two-component epoxy resin composition consisting of a base resin liquid except the following (2) and a curing agent liquid containing the following (2) or a mixture of the following (2) and (3), comprising:

(1) from 15 to 83.89% by weight of a liquid epoxy resin having 1.3 or more in weight average of epoxy groups in one molecule and an ionic conductivity of an aqueous solution obtained by extraction separation by contact



mixing with 10 times by weight of pure water at from 40 to 80°C of 20  $\mu$ s/cm or less,

(2) from 10 to 50% by weight of one or a mixture selected from (2-1) a tetrafunctional mercapto compound or (2-2) a modified polymeric mercapto derivative, having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 6  $\mu$ s/cm or less,

(3) from 0.01 to 15% by weight of a curing accelerator,

(4) from 5 to 50% by weight of an inorganic filler,

(5) from 0.1 to 5% by weight of a silane coupling agent, and

(6) from 1 to 25% by weight of rubbery polymer fine particles having a softening temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu$ m or less.

[Claim 6] A one-component sealant composition for a plastic liquid crystal cell, comprising:

(1) from 15 to 83.89% by weight of an epoxy resin having from 1.3 or more in weight average of epoxy groups in one molecule and an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to

80°C of 20  $\mu$ s/cm or less,

(2) from 10 to 50% by weight of a curing agent containing one or two or more selected from the following group from (2-3) to (2-9), having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 6  $\mu$ s/cm or less;

(2-3) a phenol novolak resin and/or an ester compound thereof

(2-4) a phenol aralkyl resin and/or an ester compound thereof

(2-5) an alicyclic compound modified novolak resin and/or an ester compound thereof

(2-6) a polycyclic aromatic compound modified novolak resin and/or an ester compound thereof

(2-7) an imidazole-epoxy adduct and/or a phenol resin salt thereof

(2-8) a micro-encapsulated imidazole compound

(2-9) a methyl methacrylate adduct of an alicyclic diamine,

(3) from 0.01 to 15% by weight of a curing accelerator,

(4) from 5 to 50% by weight of an inorganic filler,

(5) from 0.1 to 5% by weight of a silane coupling agent, and

(6) from 1 to 25% by weight of rubbery polymer fine particles having a softening temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu\text{m}$  or less.

[Claim 7] The sealant composition for a plastic liquid crystal cell of of claims 5 and 6, wherein

(I) an aqueous solution obtained by mixing the composition and 10 times by weight the composition of pure water at from 40 to 80°C has an ionic conductivity of 10  $\mu\text{S}/\text{cm}$  or less, and

a cured product of the composition has

(II) a water vapor permeability at 60°C passing a cured film of the composition having a thickness of 100  $\mu\text{m}$  of less than 200  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ ,

(III) a heat deformation temperature ( $T_g$ ) of a cured product of the composition in a range of from 0 to 100°C, and

(IV) a storage modulus under room temperature of a cured product of the composition in a range of from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa.

[Claim 8] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 7 comprising:

(V) an E type viscosity at from 50 to 100°C after

coating to a thickness of 50  $\mu\text{m}$  and being subjected to a heat treatment at from 50 to 85°C for 20 minutes of from 5 to 5,000 Pa.s.

[Claim 9] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 8, further comprising, per 100 parts by weight of the composition of any one of claims 5 to 8:

(7) from 1 to 25 parts by weight of high softening acrylic polymer fine particles having a softening temperature of 50°C or more and an average particle diameter of primary particles of 2  $\mu\text{m}$  or less.

[Claim 10] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 9, further comprising, per 100 parts by weight of the composition of any one of claims 5 to 9:

(8) from 0.1 to 5 parts by weight of wax.

[Claim 11] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 10, further comprising, per 100 parts by weight of the composition of any one of claims 5 to 10:

(9) 0.1 to 5 parts by weight of a controlling agent for providing gaps.

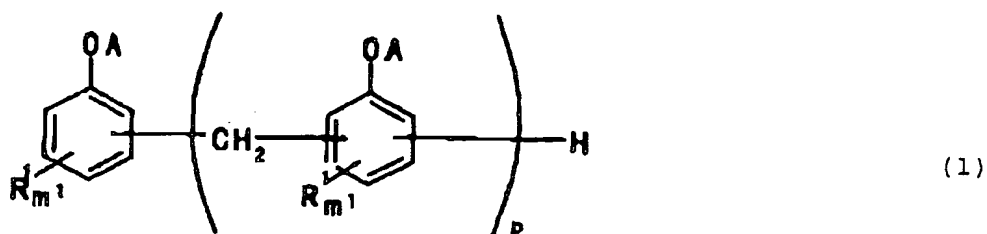
[Claim 12] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 11, wherein the epoxy resin (1) is a mixed composition of (1-1) an aliphatic and/or an alicyclic epoxy resin and (1-2) an aromatic epoxy resin.

[Claim 13] The sealant epoxy resin composition for a plastic liquid crystal display cell of any one of claims 5 to 12, wherein the aliphatic and/or alicyclic epoxy resin (1-1) is one or two or more selected from higher alcohol monoglycidyl ether, polyoxyalkylene glycol diglycidyl ether, 1,6-hexanediol diglycidyl ether, neopentyl glycol diglycidyl ether and glycerin triglycidyl ether, and the aromatic epoxy resin (1-2) is at least one or two or more selected from the group consisting of a bisphenol A type epoxy resin or an alkylene oxide adduct type epoxy resin thereof, a bisphenol F type epoxy resin or an alkylene oxide adduct type epoxy resin thereof, a bisphenol S type epoxy resin, and a bisphenol AD type epoxy resin.

[Claim 14] The sealant composition for a plastic liquid crystal cell of any one of claims 5 and 7 to 13, wherein the curing agent (2) of claim 5 consists of (2-1) solely, which is pentaerythritol tetrakis (3-mercaptopropionate).

[Claim 15] The sealant composition for a plastic liquid crystal cell of any one of claims 5 and 7 to 13, wherein the curing agent (2) of claim 5 consists of (2-2) solely, which is a modified polymercapto derivative obtained by reacting 1 equivalent in terms of active hydrogen of a polymercapto compound with from 0.01 to 0.3 equivalent in terms of active isocyanate group of a diisocyanate compound and/or an isocyanate prepolymer thereof.

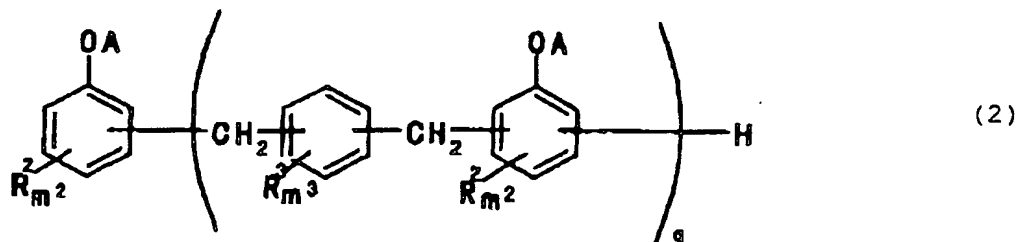
[Claim 16] The sealant composition for a plastic liquid crystal cell of any one of claims 6 to 13, wherein the curing agent (2-3) of claim 6 is a polyphenol compound or a novolak resin and/or an ester resin thereof of the following general formula (1):



wherein  $R^1$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^1$  indicates

an integer of from 1 to 3 and  $m^1$ 's may be the same or different; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit p is an integer in a range of from 1 to 100; and the repeating unit  $p=1$  represents a bisphenol derivative.

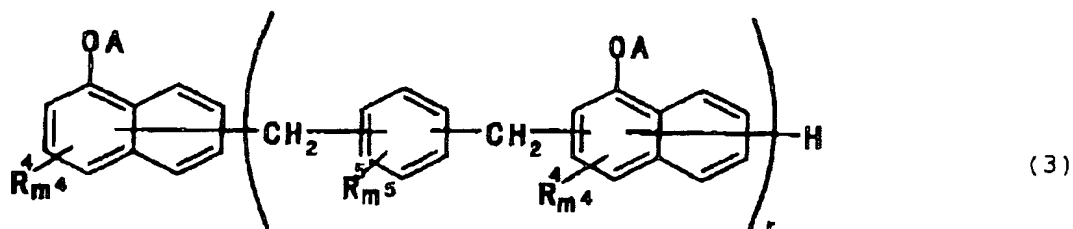
[Claim 17] The sealant composition for a plastic liquid crystal cell of any one of claims 6 to 13, wherein the curing agent (2-4) of claim 6 is a phenol aralkyl compound, a phenol aralkyl resin and/or an ester resin thereof of the following general formula (2):



wherein  $R^2$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^2$  indicates an integer of from 1 to 3 and  $m^2$ 's may be the same or different;  $R^3$  represents a hydrogen atom or a methyl group;  $m^3$  indicates an integer of from 1 to 3; A represents a

hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit q is an integer in a range of from 1 to 100; and the repeating unit q=1 represents a bisphenol derivative.

[Claim 18] The sealant composition for a plastic liquid crystal cell of any one of claims 6 to 13, wherein the curing agent (2-4) of claim 6 is a bisnaphthol compound, a naphthol type novolak resin and/or an ester resin thereof of the following general formula (3):

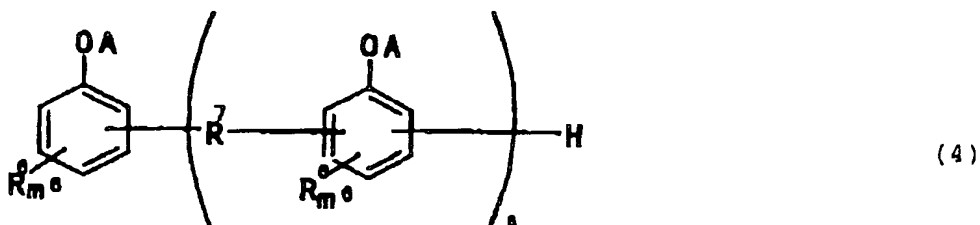


wherein  $R^4$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^4$  indicates an integer of from 1 to 3 and  $m^4$ 's may be the same or different;  $R^5$  represents a hydrogen atom or a methyl group;  $m^5$  indicates an integer of from 1 to 3; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl



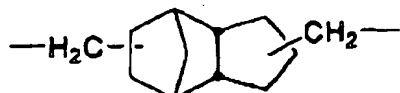
group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit  $r$  is an integer in a range of from 1 to 100; and the repeating unit  $r=1$  represents a bisnaphthol derivative.

[Claim 19] The sealant composition for a plastic liquid crystal cell of any one of claims 6 to 13, wherein the curing agent (2-5) of claim 6 is a bisnaphthol derivative, an alicyclic compound modified phenol novolak resin and/or an ester resin thereof of the following general formula (4):



wherein  $R^6$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^6$  indicates an integer of from 1 to 3 and  $m^6$ s may be the same or different;  $R^7$  represents an aliphatic ring of the following formula (6);  $A$  represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of

the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit  $s$  is an integer in a range of from 1 to 100; and the repeating unit  $s=1$  represents a bisphenol derivative.

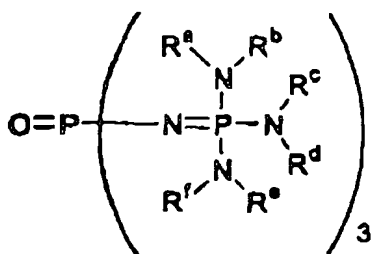


(5)



(6)

[Claim 20] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 19, wherein the curing agent (3) is a phosphine oxide compound of the following general formula (7):



(7)

wherein from  $R^a$  to  $R^f$  represent a hydrogen atom, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an aryl group with carbon number from 6 to 10, or

an aralkyl group; and Rs may be the same or different.

[Claim 21] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 20, wherein the curing agent (3) is a phosphine oxide compound of the following general formula (8):



[Claim 22] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 21, wherein the rubbery polymer fine particles (6) have a primary particle diameter of from 0.1 to 1  $\mu\text{m}$  and are crosslinked rubbery particles.

[Claim 23] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 22, wherein the high softening point acrylic polymer fine particles (7) have a softening temperature of from 60 to 150°C, from 0.1 to 5% by weight of an epoxy group, an average particle diameter of a single primary particle of 2  $\mu\text{m}$  or less, and

a micro-crosslinking structure at low temperatures within the softening temperature.

[Claim 24] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 23, wherein the component (6) is an acrylic rubber or an olefin rubber, included as a core phase of a so-called core-shell type fine particles whose shell phase is the component (7), and comprised as a composite fine particles of (6) and (7) whose weight ratio of (core:shell) is from (1:0.3) to (1:2), wherein (6) is from 1 to 15% by component conversion weight and (7) is from 0.1 to 9.5% by component conversion weight.

[Claim 25] The sealant composition for a plastic liquid crystal cell of any one of claims 5 to 24, wherein at least a part of the inorganic filler (4) is a graft product with the epoxy resin (1) and/or the silane coupling agent (5), and the graft product has a graft ratio expressed by a weight increasing rate obtained by a repeating solvent washing method of from 1 to 50 parts by weight in total of the epoxy resin (1) and the silane coupling agent (5) per 100 parts by weight of the inorganic filler (4).

[Claim 26] A process for producing a plastic liquid crystal display cell characterized in that the sealant composition for a plastic liquid crystal cell of any one of claims 1 to 25 is obtained by printing or dispenser coating on a component part of an adhering seal of a substrate for a plastic liquid crystal cell and pre-curing the substrate at a temperature of from 50 to 85°C, positioning the substrate and another substrate without the said process as a pair, a heat pressing treatment at from 60 to 100°C to adhere and fix the pair of substrates with an even thickness in a range of from 3 to 7  $\mu\text{m}$ , and charging a liquid crystal material to the inside of the cell and sealing the charging hole with a sealant composition for a two-component liquid crystal or a sealant composition for an ultraviolet cured liquid crystal.

[Claim 27] A plastic liquid crystal display cell obtained by the process for producing a plastic liquid crystal display cell of claim 26.

[DETAILED DESCRIPTION OF THE INVENTION]

[Technical Field to which the Invention Belongs]

[0001]

The present invention relates to a sealant composition for a plastic liquid crystal cell, a process for producing a plastic liquid crystal display cell, and a plastic liquid crystal display cell.

[0002]

[Prior Art]

In recent years, a light weight and thin liquid crystal display panel has been widely used as a display panel for a portable telephone. Besides, the service environment of the liquid crystal display panel is becoming severer, and therefore a high quality plastic liquid crystal display panel is demanded that has high impact resistance and high handling safety.

[0003]

Incidentally, a sealant composition for a plastic liquid crystal cell is a thermosetting resin composition used in order to form a cell sealing in a liquid crystal without a leak, and the liquid crystal fills in a space surrounded by transparent plastic substrates having transparent electrodes and an orientation film properly arranged thereon which are important as members comprised in a liquid crystal display cell.

[0004]

As a two-component thermosetting sealant composition for a plastic liquid crystal cell for a general plastic liquid crystal cell, hitherto known, for example, two-component sealant compositions have been proposed that are formed with a curing agent liquid containing a trifunctional mercapto compound and a base resin liquid containing polyethylene glycol diglycidyl ether or the like. Although these proposed sealant compositions have sufficient basic performance with respect to the sealing characteristics for a plastic liquid crystal display cell, i.e., the adhesion sealing property, the electric insulating property, the non-contamination property for liquid crystals and the like under the ordinary state, the water vapor gas barrier property, the water resistant strength characteristics and the like under a high temperature and high humidity environment, for example, at from 60 to 80 °C are considerably low. Therefore, when they are used under a high temperature and high humidity environment for a long period of time, they have a problem that moisture is penetrated into the interior of the plastic liquid crystal display cell with the lapse of time, and that as a result, fatal functional disorder of the liquid crystal display cell, typified by display unevenness, decrease in response speed and the like,

occurs at the early stage.

[0005]

Incidentally, in recent years, the demand of products using the liquid crystal display panel is being considerably increased, and in order to produce liquid crystal display panels of higher uniformity and higher quality in the production sites of those fields of art, the heat adhesion step is reviewed frequently. From the standpoint of productivity, a multiplaten heat press adhesion method has been useful, however, from the standpoint of assuring further reliability of the panel, the process for producing the liquid crystal cell needs to be reviewed.

[0006]

[Problems that the invention is to solve]

With the social background as described above, a problem to be solved is to provide a one-component or two-component sealant composition for a plastic liquid crystal display cell that is capable of being applied to not only a multiplaten heat press method but also a primary adhesion method by a sheet heat press and of producing certainly a plastic liquid crystal display panel having high seal reliability under a high temperature and high humidity environment.

[0007]



[Means of solving the problems]

The inventors found a solution of the aforementioned problem by a composition such that a specific epoxy resin, a specific curing agent, a specific curing accelerator, a specific inorganic filler, a specific silane coupling agent, and specific rubbery polymer fine particles, and further, as necessary, high-softening-point acrylic polymer fine particles, wax, and a controlling agent for providing gaps are in specific ranges, respectively, and completed the invention.

In point of fact, the invention provides the following [1] to [27].

[1] A sealant composition for a plastic liquid crystal cell, comprising:

an epoxy resin composition, wherein

(I) an aqueous solution obtained by mixing the composition and 10 times by weight of pure water at from 40 to 80°C has an ionic conductivity of 10  $\mu\text{s}/\text{cm}$  or less, and

a cured product of the composition has

(II) a water vapor permeability at 60°C passing a cured film of the composition having a thickness of 100  $\mu\text{m}$  of less than 200  $\text{g}/\text{m}^2\cdot 24\text{hrs}$ ,

(III) a heat deformation temperature ( $T_g$ ) of a cured product of the composition in a range of from 0 to 90°C,

and

(IV) a storage modulus under room temperature of a cured product of the composition in a range of from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa.

[2] The sealant composition for a plastic liquid crystal cell of [1], wherein the epoxy resin composition has

(V) an E type viscosity at from 50 to 100°C after coating to a thickness of 50  $\mu\text{m}$  and being subjected to a heat treatment at from 50 to 85°C for 20 minutes of from 5 to 5,000 Pa·s.

[3] A sealant composition for a plastic liquid crystal cell of [1] and [2], wherein the epoxy resin composition is a two-component epoxy resin composition.

[4] A sealant composition for a plastic liquid crystal cell of [1] and [2], wherein the epoxy resin composition is a one-component epoxy resin composition.

[0008]

[5] A sealant composition for a plastic liquid crystal cell, wherein the epoxy resin composition is a two-component epoxy resin composition consisting of a base resin liquid except the following (2) and a curing agent liquid containing the following (2) or a mixture of the following (2) and (3), comprising:

(1) from 15 to 83.89% by weight of a liquid epoxy

resin having 1.3 or more in weight average of epoxy groups in one molecule and an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 20  $\mu$ s/cm or less,

(2) from 10 to 50% by weight of one or a mixture selected from (2-1) a tetrafunctional mercapto compound or (2-2) a modified polymercapto derivative, having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 6  $\mu$ s /cm or less,

(3) from 0.01 to 15% by weight of a curing accelerator,

(4) from 5 to 50% by weight of an inorganic filler,

(5) from 0.1 to 5% by weight of a silane coupling agent, and

(6) from 1 to 25% by weight of rubbery polymer fine particles having a softening temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu$ m or less.

[0009]

[6] A one-component sealant composition for a plastic liquid crystal cell, comprising:

(1) from 15 to 83.89% by weight of an epoxy resin having from 1.3 or more in weight average of epoxy groups

in one molecule and an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 20  $\mu$ s/cm or less,

(2) from 10 to 50% by weight of a curing agent containing one or two or more selected from the following group from (2-3) to (2-9), having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of 6  $\mu$ s/cm or less;

(2-3) a phenol novolak resin and/or an ester compound thereof

(2-4) a phenol aralkyl resin and/or an ester compound thereof

(2-5) an alicyclic compound modified novolak resin and/or an ester compound thereof

(2-6) a polycyclic aromatic compound modified novolak resin and/or an ester compound thereof

(2-7) an imidazole-epoxy adduct and/or a phenol resin salt thereof

(2-8) a micro-encapsulated imidazole compound

(2-9) a methyl methacrylate adduct of an alicyclic diamine,

(3) from 0.01 to 15% by weight of a curing accelerator,

(4) from 5 to 50% by weight of an inorganic filler,  
(5) from 0.1 to 5% by weight of a silane coupling agent, and

(6) from 1 to 25% by weight of rubbery polymer fine particles having a softening temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu\text{m}$  or less.

[0010]

[7] The sealant composition for a plastic liquid crystal cell of [5] and [6], wherein

(I) an aqueous solution obtained by mixing the composition and 10 times by weight the composition of pure water at from 40 to 80°C has an ionic conductivity of 10  $\mu\text{S}/\text{cm}$  or less, and

a cured product of the composition has

(II) a water vapor permeability at 60°C passing a cured film of the composition having a thickness of 100  $\mu\text{m}$  of less than 200  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ ,

(III) a heat deformation temperature ( $T_g$ ) of a cured product of the composition in a range of from 0 to 100°C, and

(IV) a storage modulus under room temperature of a cured product of the composition in a range of from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa.

[8] The sealant composition for a plastic liquid

crystal cell of any one of [5] to [7] comprising:

(V) an E type viscosity at from 50 to 100°C after coating to a thickness of 50  $\mu\text{m}$  and being subjected to a heat treatment at from 50 to 85°C for 20 minutes of from 5 to 5,000 Pa·s.

[0011]

[9] The sealant composition for a plastic liquid crystal cell of any one of [5] to [8], further comprising, per 100 parts by weight of the composition of any one of [5] to [8]:

(7) from 1 to 25 parts by weight of high softening acrylic polymer fine particles having a softening temperature of 50°C or more and an average particle diameter of primary particles of 2  $\mu\text{m}$  or less.

[10] The sealant composition for a plastic liquid crystal cell of any one of [5] to [9], further comprising, per 100 parts by weight of the composition of any one of [5] to [9]:

(8) from 0.1 to 5 parts by weight of wax.

[11] The sealant composition for a plastic liquid crystal cell of any one of [5] to [10], further comprising, per 100 parts by weight of the composition of any one of [5] to [10]:

(9) 0.1 to 5 parts by weight of a controlling agent for providing gaps.

[0012]

[12] The sealant composition for a plastic liquid crystal cell of any one of [5] to [11], wherein the epoxy resin (1) is a mixed composition of (1-1) an aliphatic and/or an alicyclic epoxy resin and (1-2) an aromatic epoxy resin.

[13] The sealant epoxy resin composition for a plastic liquid crystal display cell of any one of [5] to [12], wherein the aliphatic and/or alicyclic epoxy resin (1-1) is one or two or more selected from higher alcohol monoglycidyl ether, polyoxyalkylene glycol diglycidyl ether, 1,6-hexanediol diglycidyl ether, neopentyl glycol diglycidyl ether and glycerin triglycidyl ether, and the aromatic epoxy resin (1-2) is at least one or two or more selected from the group consisting of a bisphenol A type epoxy resin or an alkylene oxide adduct type epoxy resin thereof, a bisphenol F type epoxy resin or an alkylene oxide adduct type epoxy resin thereof, a bisphenol S type epoxy resin, and a bisphenol AD type epoxy resin.

[14] The sealant composition for a plastic liquid crystal cell of any one of [5] and [7] to [13], wherein the curing agent (2) of [5] consists of (2-1) solely, which is pentaerythritol tetrakis (3-mercaptopropionate).

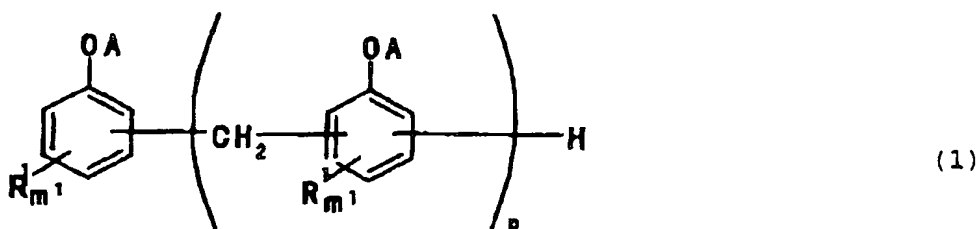
[0013]

[15] The sealant composition for a plastic liquid

crystal cell of any one of [5] and [7] to [13], wherein the curing agent (2) of [5] consists of (2-2) solely, which is a modified polymericthio derivative obtained by reacting 1 equivalent in terms of active hydrogen of a polymericthio compound with from 0.01 to 0.3 equivalent in terms of active isocyanate group of a diisocyanate compound and/or an isocyanate prepolymer thereof.

[16] The sealant composition for a plastic liquid crystal cell of any one of [6] to [13], wherein the curing agent (2-3) of claim 6 is a polyphenol compound or a novolak resin and/or an ester resin thereof of the following general formula (1):

[0014]



[0015]

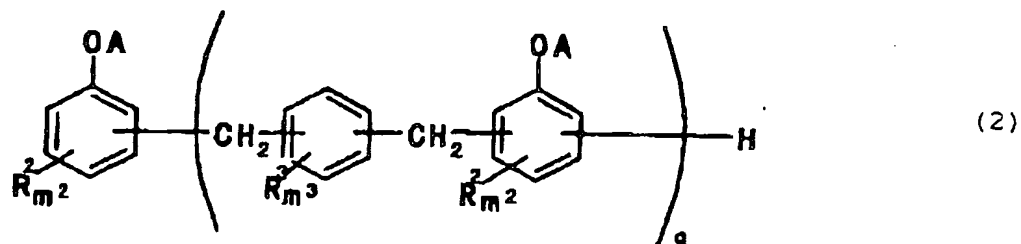
(wherein  $\text{R}^1$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^1$  indicates an integer of from 1 to 3 and  $m^1$ 's may be the



same or different; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit p is an integer in a range of from 1 to 100; and the repeating unit p=1 represents a bisphenol derivative.)

[17] The sealant composition for a plastic liquid crystal cell of any one of [6] to [13], wherein the curing agent (2-4) of [6] is a phenol aralkyl compound, a phenol aralkyl resin and/or an ester resin thereof of the following general formula (2):

[0016]



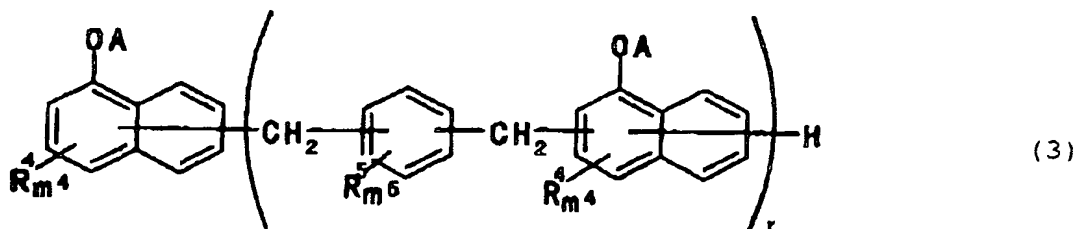
[0017]

(wherein  $R^2$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^2$  indicates an integer of from 1 to 3 and  $m^2$ 's may be the same or different;  $R^3$  represents a hydrogen atom or a methyl group;  $m^3$  indicates an integer of from 1 to 3; A

represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit  $q$  is an integer in a range of from 1 to 100; and the repeating unit  $q=1$  represents a bisphenol derivative.)

[18] The sealant composition for a plastic liquid crystal cell of any one of [6] to [13], wherein the curing agent (2-4) of [6] is a bisnaphthol compound, a naphthol type novolak resin and/or an ester resin thereof of the following general formula (3):

[0018]



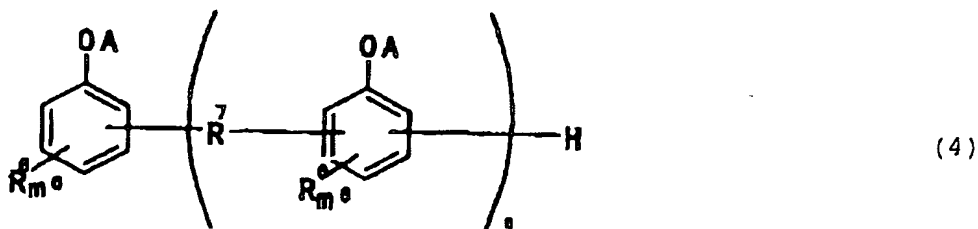
[0019]

(wherein  $R^4$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^4$  indicates an integer of from 1 to 3 and  $m^4$ 's may be the same or different;  $R^5$  represents a hydrogen atom or a

methyl group;  $m^5$  indicates an integer of from 1 to 3; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit r is an integer in a range of from 1 to 100; and the repeating unit  $r=1$  represents a bisnaphthol derivative.)

[19] The sealant composition for a plastic liquid crystal cell of any one of [6] to [13], wherein the curing agent (2-5) of [6] is a bisnaphthol derivative, a alicyclic compound modified phenol novolak resin and/or an ester resin thereof of the following general formula (4):

[0020]



[0021]

(wherein  $R^6$  represents a hydrogen atom, a halogen atom, an acyloxy group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^6$  indicates an integer of from 1 to 3 and  $m^6$ s may be the

same or different;  $R^7$  represents an aliphatic ring of the following formula (6); A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit  $s$  is an integer in a range of from 1 to 100; and the repeating unit  $s=1$  represents a bisphenol derivative.)

[0022]



(5)

[0023]

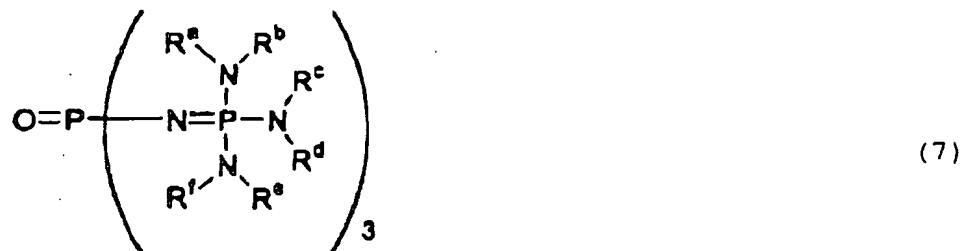


(6)

[0024]

[20] The sealant composition for a plastic liquid crystal cell of any one of [5] to [19], wherein the curing agent (3) is a phosphine oxide compound of the following general formula (7):

[0025]

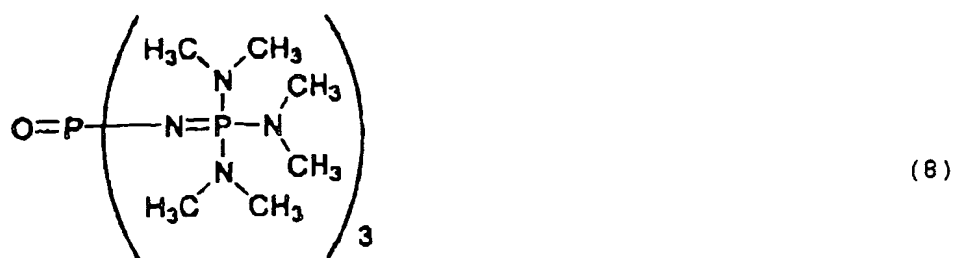


[0026]

(wherein from  $\text{R}^a$  to  $\text{R}^e$  represent a hydrogen atom, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an aryl group with carbon number from 6 to 10, or an aralkyl group; and Rs may be the same or different.)

[21] The sealant composition for a plastic liquid crystal cell of any one of [5] to [20], wherein the curing agent (3) is a phosphine oxide compound of the following general formula (8):

[0027]



[0028]

[22] The sealant composition for a plastic liquid

crystal cell of any one of [5] to [21], wherein the rubbery polymer fine particles (6) have a primary particle diameter of from 0.1 to 1  $\mu\text{m}$  and are crosslinked rubbery particles.

[23] The sealant composition for a plastic liquid crystal cell of any one of [5] to [22], wherein the high softening point acrylic polymer fine particles (7) have a softening temperature of from 60 to 150°C, from 0.1 to 5% by weight of an epoxy group, an average particle diameter of a single primary particle of 2  $\mu\text{m}$  or less, and a micro-crosslinking structure at low temperatures within the softening temperature.

[24] The sealant composition for a plastic liquid crystal cell of any one of [5] to [23], wherein the component (6) is an acrylic rubber or an olefin rubber, included as a core phase of a so-called core-shell type fine particles whose shell phase is the component (7), and comprised as a composite fine particles of (6) and (7) whose weight ratio of (core:shell) is from (1:0.3) to (1:2), wherein (6) is from 1 to 15% by component conversion weight and (7) is from 0.1 to 9.5% by component conversion weight.

[0029]

[25] The sealant composition for a plastic liquid crystal cell of any one of [5] to [24], wherein at least a

part of the inorganic filler (4) is a graft product with the epoxy resin (1) and/or the silane coupling agent (5), and the graft product has a graft ratio expressed by a weight increasing rate obtained by a repeating solvent washing method of from 1 to 50 parts by weight in total of the epoxy resin (1) and the silane coupling agent (5) per 100 parts by weight of the inorganic filler (4).

[26] A process for producing a plastic liquid crystal display cell characterized in that the sealant composition for plastic liquid crystal cell of any one of [1] to [25] is obtained by printing or dispenser coating on a component part of an adhering seal of a substrate for a plastic liquid crystal cell and pre-curing the substrate at a temperature of from 50 to 85°C, positioning the substrate and another substrate without the said process as a pair, a heat pressing treatment at from 60 to 100°C to adhere and fix the pair of substrates with an even thickness in a range of from 3 to 7  $\mu\text{m}$ , and charging a liquid crystal material to the inside of the cell and sealing the charging hole with a sealant composition for a two-component liquid crystal or a sealant composition for an ultraviolet cured liquid crystal.

[27] A plastic liquid crystal display cell obtained by the process for producing a plastic liquid crystal display cell of [26].

[0030]

[Embodiment of the invention]

The sealant composition for a plastic liquid crystal display cell of the invention is a sealant composition for plastic liquid crystal consisting of an epoxy resin composition and simultaneously assures the following functionalities (I) to (IV).

(I) An aqueous solution obtained by mixing the composition and 10 times by weight the composition of pure water at from 40 to 80°C has an ionic conductivity of 10  $\mu\text{S}/\text{cm}$  or less.

(II) A water vapor permeability at 60°C passing a cured film of the composition having a thickness of 100  $\mu\text{m}$  is less than 200  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ .

(III) A heat deformation temperature ( $T_g$ ) of a cured product of the composition is in a range of from 0 to 99°C.

(IV) A storage modulus under room temperature of a cured product of the composition in a range is from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa.

[0031]

In the sealant composition for a plastic liquid crystal cell of the invention, as an index of a concentration of free ions derived from the composition, it is preferred that an aqueous solution obtained by mixing the composition and 10 times by weight the



composition of pure water at from 40 to 80°C has an ionic conductivity within the above-mentioned range. The ionic conductivity should be 10 $\mu$ s/cm or less in order that maintenance of the long-term display functionality of the resulting liquid crystal display cell can be assured.

[0032]

The following is the reason that the property of a permeability at 80°C as an index of a water vapor gas barrier of the sealant composition for a plastic liquid crystal cell is provided as the foregoing. That is to say, in case where a water vapor permeability at 60°C passing a cured film of the sealant composition for a plastic liquid crystal cell having a thickness of 100  $\mu$ m at 60°C and relative humidity of 95% per 24 hours is more than 200 g/m<sup>2</sup>·24hrs, moisture is penetrated into the interior of the plastic liquid display cell in a short time, and as a result, fatal functional disorder of the liquid crystal display cell, such as display unevenness, decrease in response speed and the like, occurs. More preferably, in the sealant composition for a plastic liquid crystal of the invention, the water vapor permeability at 60°C of the cured product is 150 g/m<sup>2</sup>·24hrs or less, and further preferably less than 100 g/m<sup>2</sup>·24hrs.

[0033]

As the reason that heat deformation temperature (T<sub>g</sub>)

of a cured product of the sealant composition for a plastic liquid crystal cell measured by TMA (thermomechanical analysis) as an index relative to heat resistant rigidity property is provided as the foregoing, it is preferred that the heat deformation temperature ( $T_g$ ) of the cured product measured by TMA is  $0^{\circ}\text{C}$  or more in order that the display reliability critical temperature for a long period of time of the liquid crystal display cell can be improved. Besides, it is preferably  $99^{\circ}\text{C}$  or less, and according to the configuration, the impact resistance and the heat resistant adhesion reliability of the cell can be practically assured. It is more preferably in a range of from  $25$  to  $99^{\circ}\text{C}$ , and further preferably in a range of from  $50$  to  $99^{\circ}\text{C}$ .

[0034]

As the reason that the storage modulus under room temperature as a rigid index of a cured product in a normal state of the sealant composition for a plastic liquid crystal cell is provided as the foregoing, it is preferred that the storage modulus of a cured product itself under room temperature is in a range of from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa in order that the production yield of the resulting plastic liquid crystal display cell is improved with practical advantage. In the sealant composition of a plastic liquid crystal cell, the storage modulus of a

cured product is more preferably in a range of from  $0.8 \times 10^4$  to  $0.9 \times 10^6$  Pa, and further preferably in a range of from  $1 \times 10^4$  to  $0.8 \times 10^6$  Pa.

[0035]

It is more preferred that the sealant composition for a plastic liquid crystal cell of the invention is a composition that assures the foregoing (I) to (IV) and also the following (V). (V) is an E type viscosity at from 50 to 100°C after coating to a thickness of 50  $\mu\text{m}$  and being subjected to a heat treatment at from 50 to 85°C for 20 minutes (hereinafter referred to as the B stage composition) is in a range of from 5 to 5,000 Pa·s.

[0036]

The following is the reason that as a property relative to the sealant composition for a plastic liquid crystal cell, in the composition subjecting to B stage at a temperature of whole range of from 50 to 100°C, the E type viscosity is preferably provided as the foregoing. That is to say, when in the composition subjecting to B stage at a temperature in whole range of from 50 to 100°C, the E type viscosity of the sealant composition for a plastic liquid crystal cell exceeds 5 Pa·s, formation of seal leak upon heat press adhesion by multiplaten heat press or sheet heat press can be practically suppressed. When at a temperature of whole range of from 50 to 100°C,

the E type viscosity is 5,000 Pa·s or less, desired gap control can be practically ensured upon heat press adhesion by multiplaten heat press or sheet heat press. In the sealant composition for a plastic liquid crystal cell of the invention, as a property of the composition subjected to B stage, the E type viscosity is more preferably in a range of from 5 to 3,000 Pa·s, and further preferably in a range of from 5 to 1,000 Pa·s.

[0037]

It is preferable that the sealant composition for a plastic liquid crystal cell of the invention is an epoxy resin composition of either of one-component and two-component type.

[0038]

The sealant composition for a plastic liquid crystal display cell of the invention basically contains (1) an epoxy resin having 1.7 or more in weight average of epoxy groups in one molecule and an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water of 20  $\mu$ s/cm or less [hereinafter occasionally referred to simply as the epoxy resin (1)], (2) a curing agent having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of at the most 6

μs/cm or less [hereinafter occasionally referred to simply as the curing agent (2)], (3) a curing accelerator, (4) an inorganic filler, (5) a silane coupling agent, (6) rubbery polymer fine particles having a softening point temperature of 0°C or less and an average particle diameter of primary particles of 5 μm or less, depending on necessity, (7) high softening point acrylic polymer fine particles having a softening point temperature of 50°C or more and an average particle diameter of primary particles thereof of 2 μm or less, (8) wax, (9) a controlling agent for providing gaps, a solvent, a leveling agent, a pigment, a dye, a plasticizer, a defoaming agent, other additives and the like. The constitutional components will be described.

[0039]

[(1) Epoxy Resin]

The epoxy resin (1) used for the invention is a resin having 1.3 or more, in terms of weight average, of epoxy groups per one molecule, an ionic conductivity of an aqueous solution obtained by extraction by mixing 10 times by weight of pure water at from 40 to 80°C of 20 μm/cm or less, preferably 1.4 or more, in terms of weight average, of epoxy groups per one molecule, and more preferably from 1.5 to 3, in terms of weight average. These resins may be used solely or by mixing different resins.

It is preferred that the epoxy resin (1) has 1.3 or more, in terms of weight average, of epoxy groups per one molecule to improve heat resistance.

The epoxy groups have no particular bounds when the epoxy groups are an epoxy resin having a stipulated epoxy resin or a mixture thereof, and a mixture of a monofunctional epoxy resin and a polyfunctional epoxy resin, or sole or a mixture of a polyfunctional epoxy resin can be used. Besides, a modified epoxy resin thereof can be preferably used. Not in order to put particular bounds, the epoxy resin in the sealant composition for a plastic liquid crystal cell can be fractionated by liquid chromatography, and the number of functional groups per one molecule of the epoxy resin can be determined by the epoxy equivalent and the weight average molecule weight.

[0040]

[Monofunctional Epoxy Resin]

Examples of the monofunctional epoxy resin used for the invention include an aliphatic monoglycidyl ether compound, a alicyclic monoglycidyl ether compound, an aromatic monoglycidyl ether compound, an aliphatic monoglycidyl ester compound, an aromatic monoglycidyl ester compound, an alicyclic monoglycidyl ester compound, a nitrogen-containing monoglycidyl ether compound, a

monoglycidylpropylpolysiloxane compound, a monoglycidylalkane and the like. Other monofunctional epoxy resins may be used.

[0041]

(Aliphatic Monoglycidyl Ether Compound)

Examples include an aliphatic monoglycidyl ether compound obtained by the reaction of a polyoxyalkylene monoalkyl ether having an alkyl group whose carbon atoms are represented by a integer of from 1 to 6 or an alkenyl group with epichlorohydrin, an aliphatic monoglycidyl ether compound obtained by the reaction of a higher alcohol and epichlorohydrin, and the like.

[0042]

Examples of a polyoxyalkylene glycol monoalkyl ether having an alkyl group whose carbon atoms are represented by a integer of from 1 to 6 or an alkenyl group include ethylene glycol monoalkyl ether, diethylene glycol monoalkyl ether, triethylene glycol monoalkyl ether, polyethylene glycol monoalkyl ether, propylene glycol monoalkyl ether, dipropylene glycol monoalkyl ether, tripropylene glycol monoalkyl ether, polypropylene glycol monoalkyl ether and the like.

[0043]

Examples of the higher alcohol include n-butanol, isobutanol, n-octanol, 2-ethylhexyl alcohol,

dimethylolpropane monoalkyl ether, trimethylolpropane dialkyl ether, glycerin dialkyl ether, dimethylolpropane monoalkyl ester, trimethylolpropane dialkyl ester, glycerin dialkyl ester and the like.

[0044]

(Alicyclic Monoglycidyl Ether Compound)

Examples include an alicyclic monoglycidyl ether compound obtained by the reaction of an alicyclic alcohol having an alicyclic alkyl group whose carbon atoms are represented by a integer of from 6 to 9 with epichlorohydrin, and the like. Examples of the alicyclic alcohol to use for the reaction include cyclohexanol and the like.

[0045]

(Aromatic Monoglycidyl Ether Compound)

Examples include an aromatic monoglycidyl ether compound obtained by the reaction of an aromatic alcohol with epichlorohydrin, and the like. Examples of the aromatic alcohol to use for the reaction include phenol, methylphenol, ethylphenol, n-propylphenol, isopropylphenol, n-butylphenol, benzyl alcohol, t-butylphenol, xyleneol, naphthol and the like.

[0046]

(Aliphatic or Aromatic Monoglycidyl Ester Compound)

Examples include an aliphatic monoglycidyl ester



compound or an aromatic monoglycidyl ester compound obtained by the reaction of a monoalkyl aliphatic dicarboxylate ester or a monoalkyl aromatic dicarboxylate ester with epichlorohydrin, and the like.

[0047]

[Polyfunctional Epoxy Resin]

The polyfunctional epoxy resin means an epoxy resin which normally contains from 2 to 4 of epoxy groups in one molecule on average, and also a resin containing more epoxy groups as far as the resin does not obstruct the effect of the invention. Examples of the polyfunctional epoxy resin include an aliphatic polyglycidyl ether compound, an aromatic polyglycidyl ether compound, a trisphenol type polyglycidyl ether compound, a hydroquinone type polyglycidyl ether compound, a resorcinol type polyglycidyl ether compound, an aliphatic polyglycidyl ester compound, an aromatic polyglycidyl ester compound, an aliphatic polyglycidyl ether ester compound, an aromatic polyglycidyl ether ester compound, an alicyclic polyglycidyl ether compound, an aliphatic polyglycidyl amine compound, an aromatic polyglycidyl amine compound, a hydantoin type polyglycidyl compound, a biphenyl type polyglycidyl compound, a novolak type polyglycidyl ether compound, an epoxidated diene polymer and the like. Other polyfunctional epoxy resins may be

used.

[0048]

(Aliphatic Polyglycidyl Ether Compound).

Examples include aliphatic polyglycidyl ether compounds obtained by the reaction of a polyoxyalkylene glycol or a polyalcohol with epichlorohydrin, and the like.

Examples of the polyoxyalkylene glycol to use for the reaction include ethylene glycol, polyoxyethylene glycol, propylene glycol, polyoxypropylene glycol, tetramelen glycol, polyoxytetramethylene glycol and the like.

[0049]

Examples of the polyalcohol to use for the reaction include ,1,6-hexanediol, neopentyle glycol, trimethylolpropane, spiroglycol, glycerin, alkylene oxide adduct of the following aromatic diol and the like.

[0050]

(Aromatic Polyglycidyl Ether Compound)

Examples include aromatic polyglycidyl ether compounds obtained by the reaction of an aromatic diol with epichlorohydrin, and the like.

Examples of the aromatic diol to use for the reaction include bisphenol A or an alkylene adduct thereof, bisphenol S or an alkylene adduct thereof, bisphenol F or an alkylene adduct thereof, bisphenol AD or an alkylene

adduct thereof and the like.

[0051]

(Trisphenol Type Polyglycidyl Ether Compound)

Examples include trisphenol type polyglycidyl ether compounds obtained by the reaction of a trisphenol with epichlorohydrin.

[0052]

Examples of the trisphenol to use for the reaction include 4,4',4''-methylidenetrisphenol, 4,4',4''-methylidenetris(2-methylphenol), 4,4'-((2-hydroxyphenyl)methylene)-bis(2,3,6-trimethylphenol), 4,4',4''-ethylidenetrisphenol, 4,4'-((2-hydroxyphenyl)methylene)bis(2-methylphenol), 4,4'-((2-hydroxyphenyl)ethylene)bis(2-methylphenol), 4,4'-((4-hydroxyphenyl)methylene)bis(2-methylphenol), 4,4'-((4-hydroxyphenyl)ethylene)bis(2-methylphenol), 4,4'-((2-hydroxyphenyl)methylene)bis(2,6-dimethylphenol), 4,4'-((2-hydroxyphenyl)ethylene)bis(2,6-dimethylphenol), 4,4'-((4-hydroxyphenyl)methylene)bis(2,6-dimethylphenol), 4,4'-((4-hydroxyphenyl)ethylene)bis(2,6-dimethylphenol), 4,4'-((2-hydroxyphenyl)methylene)bis(3,5-dimethylphenol), 4,4'-((2-hydroxyphenyl)ethylene)bis(3,5-dimethylphenol), 4,4'-((3-hydroxyphenyl)methylene)-bis(2,3,6-trimethylphenol), 4,4'-((4-hydroxyphenyl)methylene)-bis(2,3,6-trimethylphenol), 4,4'-((2-hydroxyphenyl)methylene)bis(2-cyclohexyl-5-

methylphenol), 4,4'-((3-hydroxyphenyl)methylene)bis(2-cyclohexyl-5-methylphenol), 4,4'-((4-hydroxyphenyl)methylene)bis(2-cyclohexyl-5-methylphenol), 4,4'-(1-(4-(1-(4-hydroxyphenyl)-1-methylethyl)phenoylethylidene)bisphenol), 4,4'-((3,4-dihydroxyphenyl)methylene)bis(2-methylphenol), 4,4'-((3,4-dihydroxyphenyl)methylene)-bis(2,6-dimethylphenol), 4,4'-((3,4-dihydroxyphenyl)methylene)-bis(2,3,6-trimethylphenol), 4-(bis(3-cyclohexyl-4-hydroxy-6-methylphenyl)methyl)-1,2-benzenediol and the like.

[0053]

(Hydroquinone Type Polyglycidyl Ether Compound)

Examples include hydroquinone type polyglycidyl ether compounds obtained by the reaction of hydroquinone with epichlorohydrin, and the like.

[0054]

(Resorcinol Type Polyglycidyl Ether Compound)

Examples include resorcinol type polyglycidyl ether compounds obtained by the reaction of resorcinol with epichlorohydrin, and the like.

[0055]

(Aliphatic Polyglycidyl Ester Compound)

Examples include aliphatic polyglycidyl ester compounds obtained by the reaction of an aliphatic dicarboxylic acid, represented by adipic acid, with

epichlorohydrin, and the like.

[0056]

(Aromatic Polyglycidyl Ester Compound)

Examples include aromatic polyglycidyl ester compounds obtained by the reaction of an aromatic dicarboxylic acid with epichlorohydrin, and the like.

Examples of the aromatic dicarboxylic acid to use for the reaction include isophthalic acid, terephthalic acid, pyromellitic acid and the like.

[0057]

(Aliphatic or Aromatic Polyglycidyl Ether Ester Compound)

Examples include aliphatic polyglycidyl ether ester compounds or aromatic polyglycidyl ether ester compounds obtained by the reaction of a hydroxydicarboxylic acid compound with epichlorohydrin, and the like.

[0058]

(Alicyclic Polyglycidyl Ether Compound)

Examples include alicyclic polyglycidyl ether compounds represented by a dicyclopentadiene type polyglycidyl ether compound and the like.

[0059]

(Aliphatic Polyglycidyl Amine Compound)

Examples include aliphatic polyglycidyl amine compounds obtained by the reaction of an aliphatic amine,

represented by ethylenediamine, with epichlorohydrin, diethylenetriamine, triethylene tetramine and the like.

[0060]

(Aromatic Polyglycidyl Amine Compound)

Examples include aromatic polyglycidyl amine compounds obtained by the reaction of an aromatic diamine, represented by diaminodiphenylmethane, aniline, metaxylylenediamine and the like, with epichlorohydrin, and the like.

[0061]

(Hydantoin Type Polyglycidyl Compound)

Examples include hydantoin type polyglycidyl compounds obtained by the reaction of hydantoin and a derivative thereof with epichlorohydrin, and the like.

[0062]

(Novolak Type Polyglycidyl Ether Compound)

Examples include novolak type polyglycidyl ether compounds obtained by the reaction of a novolak resin derived from an aromatic alcohol, represented by phenol, cresol, naphthol and the like, and formaldehyde with epichlorohydrin, and the like. A modified phenol novolak resin obtained by the reaction of a modified phenol resin formed by combining, with a methylene bond, a phenol nucleus derived from phenol and p-xylylene dichloride, and a paraxylene nucleus, with epichlorohydrin, and the like

is also included.

(Epoxidated Diene Polymer)

Examples include epoxidated polybutadiene, epoxidated polyisoprene and the like.

[0063]

It is preferred that the epoxy resin (1) has the ionic conductivity of an aqueous solution obtained by mixing extraction of the single or a mixture of different resins and 10 times by weight of pure water at from 40 to 80°C of 20  $\mu$ s/cm or less, preferably 15 $\mu$ s/cm or less, and more preferably 5 $\mu$ s/cm or less, in order that upon contacting a cured product of the sealant composition for a plastic liquid crystal cell of the invention with a liquid crystal, migration of free ions to the liquid crystal phase can be inhibited. Two or more of different resins can be used when the foregoing requirements as an index of a total content of free ions in the mixture are fulfilled.

[0064]

It is preferred that the epoxy resin (1) is a mixed composition of an aliphatic and/or alicyclic epoxy resin (1-1) and an aromatic epoxy resin (1-2). Expressing a mixing weight ratio thereof by (1-1) : (1-2), the mixing weight ratio is preferably in a range of (5 : 95) to (70 : 30), and particularly preferably (10 : 90) to (40 : 60).

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It is preferred that the aliphatic and/or alicyclic epoxy resin (1-1) is one or two or more selected from among a higher alcohol monoglycidyl ether group, an alkylene glycol monoglycidyl ether group, an alkylene glycol diglycidyl ether group, a polyoxyalkylene glycol monoglycidyl ether group having a repetition polymerization degree represented by an integral number of 1 to 50, a polyoxyalkylene glycol diglycidyl ether group having a repetition polymerization degree represented by an integral number of 1 to 50, 1,6-hexane diglycidyl ether, glycerin diglycidyl ether, glycerin triglycidyl ether, neopentyl glycol diglycidyl ether, trimethylolpropane triglycidyl ether and the like and modified epoxy resins thereof. It is more preferred that the aliphatic and/or alicyclic epoxy resin (1-1) is one or two or more selected from among higher alcohol monoglycidyl ether, polyoxyalkylene glycol diglycidyl ether, 1,6-hexanediol diglycidyl ether, neopentyl glycol diglycidyl ether, and glycerin triglycidyl ether.

[0065]

Besides, it is preferred that the aromatic epoxy resin (2-1) is at least a resin or a mixture thereof selected from a group of a bisphenol A type epoxy resin or an alkylene oxide adduct type epoxy resin thereof, a bisphenol F type epoxy resin or an alkylene oxide adduct



type epoxy resin thereof, a bisphenol S type epoxy resin, and a bisphenol AD type epoxy resin.

[0066]

The polystyrene conversion weight average molecular weight of the epoxy resin (1) obtained by gel permeation chromatography (GPC) is preferably 7,000 or less, more preferably from 150 to 5,000, and further preferably from 350 to 3,500.

[0067]

When the polystyrene conversion weight average molecular weight is 7,000 or less, the E type viscosity after subjecting to B stage of the sealant composition for a plastic liquid crystal cell can be 5,000 Pa·s or less, which ensures suitability of sheet press heat adhesion. Furthermore, it is preferred that the polystyrene conversion weight average molecular weight is 150 or more, in order to keep the cross-linking concentration of the obtained cured product high and ensure reliability of the heat-resistant sealant.

[0068]

From 15 to 83.89% by weight, preferably from 25 to 70% by weight, of the epoxy resin (1) is comprised in the sealant composition for a plastic liquid crystal cell.

Besides, the following epoxy resin (1) may be purified or have high purity to satisfy the foregoing

requirements by the known purification. Not in order to put particular bounds, but examples include a purification method by water washing and solvent extraction, an ultra filtration method, a distillation purification method and the like.

[0069]

[(2) Curing Agent]

As for the curing agent (2) used in the sealant composition for a plastic liquid crystal cell of the invention, a curing agent (2) used in the case where the sealant composition for a plastic liquid crystal cell is two-component type is one or two selected from the following (2-1) and (2-2) having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of at the most 6  $\mu\text{s}/\text{cm}$  or less. A curing agent (2) used in the case where the sealant composition for a plastic liquid crystal cell of the invention is one-component type is one or two or more selected from the following from (2-3) to (2-9) having an ionic conductivity of an aqueous solution obtained by extraction separation by contact mixing with 10 times by weight of pure water at from 40 to 80°C of at the most 6  $\mu\text{s}/\text{cm}$  or less.

(2-1) tetrafunctional mercapto compound

(2-2) modified polymeric mercapto derivative

(2-3) phenol novolak resin and/or an ester compound thereof

(2-4) phenol aralkyl resin and/or an ester compound thereof

(2-5) alicyclic compound modified novolak resin and/or an ester compound thereof

(2-6) polycyclic aromatic compound modified novolak resin and/or an ester compound thereof

(2-7) imidazole-epoxy adduct and/or a phenol resin salt thereof

(2-8) micro-encapsulated imidazole compound

(2-9) methyl methacrylate adduct of an alicyclic diamine

[0070]

The following is an explanation of from (2-1) to (2-9).

(2-1) Tetrafunctional Mercapto Compound

Not in order to put particular bounds, but a tetrafunctional mercapto compound capable of being handled as a liquid at room temperature may be preferably used. Concrete examples include pentaerythritol tetrakis (2-mercaptoacetate), pentaerythritol tetrakis (3-mercaptopropionate), pentaerythritol tetrakis (2-isopropionate) and the like. It is preferred that pentaerythritol tetrakis (3-mercaptopropionate) is used

solely.

[0071]

Examples of the modified polymercapto derivative include those obtained by reacting 1 equivalent in terms of active hydrogen of a bifunctional to tetrafunctional mercapto compound with from 0.01 to 0.3 equivalent, preferably from 0.05 to 0.25 equivalent, and further preferably from 0.1 to 0.2 equivalent, in terms of active isocyanate groups of a diisocyanate compound and/or a isocyanate prepolymer, or an epoxy resin modified polymercapto derivative and the like obtained by reacting 1 equivalent in terms of active hydrogen of a bifunctional to tetrafunctional mercapto compound with from 0.01 to 0.3 equivalent, preferably from 0.05 to 0.25 equivalent, and further preferably from 0.1 to 0.2 equivalent, in terms of active glycidyl of the epoxy resin. It is further preferred that the curing agent (2) consists of (2-2) solely and that (2-2) is a modified polymercapto derivative obtained by reacting 1 equivalent in terms of active hydrogen of a polymercapto compound with from 0.01 to 0.3 equivalent in terms of active isocyanate groups of a diisocyanate compound and/or a isocyanate prepolymer thereof.

[0072]

Examples of the bifunctional mercapto compound

include ethylene glycol dimercaptopropionate, polyoxyethylene glycol dimercaptopropionate, propylene glycol dimercaptopropionate, polypropylene glycol dimercaptopropionate, tetramethylene glycol dimercaptopropionate, polyoxytetramethylene glycol dimercaptopropionate, neopentyl glycol dimercaptopropionate and the like.

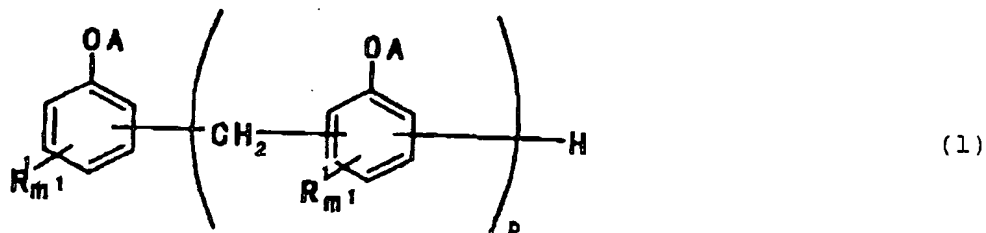
[0073]

Examples of the trifunctional mercapto compound include glycerin trimercaptopropionate, trimethylolpropane trimercaptopropionate, trismercaptopisocyanurate and the like. The tetrafunctional mercapto compound may be one or two or more selected from (2-1).

[0074]

Examples of the diisocyanate compound has no particular bounds but include trilene diisocyanate, diphenylmethane diisocyanate, isophorone diisocyanate, norbornane diisocyanate, hexamethylene diisocyanate and the like. And examples of the isocyanate prepolymer preferably include a liquid prepolymer and the like at from room temperature to 50°C having biterminal isocyanate groups whose polystyrene conversion weight average molecular weight by GPC derived from the diisocyanate and the polyol is 50000 or less.

[0075]



[0076]

(wherein  $R^1$  represents a hydrogen atom, a halogen atom, an hydroxyl group, a liner, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^1$  indicates an integer of from 1 to 3 and  $m^1$ 's may be the same or different; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit p is an integer in a range of from 1 to 100; and the repeating unit  $p=1$  represents a bisphenol derivative.)

[0077]

It is preferred that the esterified phenol novolak resin is an esterified phenol novolak resin such that 10 to 100 mol% of inherent phenolic hydroxyl groups of a phenol novolak resin made by subjecting phenol groups described below and formaldehyde to addition condensation under acidity is further subjected to aromatic and/or

aliphatic acylation denaturation, for example. More preferably, as the (2-1), it is desirable to select and use the esterified phenol novolak resin having a softening point temperature of 75°C or more, and further preferably, one having a polystyrene conversion weight average molecular weight obtained by GPC in a range of 300 to 10000, and particularly preferably in a range of 500 to 7500.

[0078]

Preferable examples of the phenol include one or two or more selected from phenol, cresol, xlenol, hydroquinone, methyl hydroquinone, catechol, resorcin, ethyl phenol, diethyl phenol, n-propyl phenol, isopropyl phenol, n-butyl phenol, t-butyl phenol, 2-ethylhexyl phenol, phenyl phenol and the like. Further preferable examples are phenol and/or cresol.

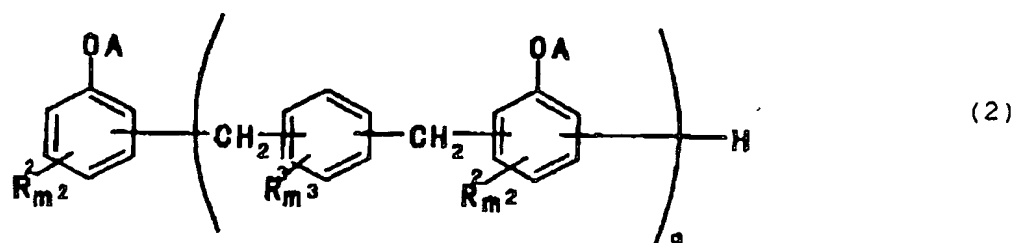
[0079]

(2-4) Phenol Aralkyl Resin and/or Esterified  
Composition thereof

It is expressed by an esterified phenol aralkyl resin expressed by a general formula (2) described below, and although not a particular restriction, a good example is an esterified phenol aralkyl resin such that 10 to 100 mol% of inherent phenolic hydroxyl groups of a phenol aralkyl resin made by being derived from phenol groups and

a xylylene dichloride compound or a xylylene dialkyl ether compound in the presence of a Friedel-Crafts catalyst and subjecting free phenol to vacuum elimination to 0.01% by weight or less is further subjected to aromatic and/or aliphatic acylation denaturation, for example.

[0080]



[0081]

(wherein  $R^2$  represents a hydrogen atom, a halogen atom, an hydroxyl group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^2$  indicates an integer of from 1 to 3 and  $m^2$ 's may be the same or different;  $R^3$  represents a hydrogen atom or a methyl group;  $m^3$  indicates an integer of from 1 to 3; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit  $q$  is an integer in a range of from 1 to 100; and the repeating unit  $q=1$  represents a bisphenol



derivative.)

[0082]

Preferable examples of the phenol include one or two or more selected from phenol, cresol, xylenol, hydroquinone, methyl hydroquinone, catechol, resorcin, ethyl phenol, diethyl phenol, n-propyl phenol, isopropyl phenol, n-butyl phenol, t-butyl phenol, 2-ethylhexyl phenol, phenyl phenol and the like. Further preferable examples are phenol and/or cresol.

[0083]

Examples of the xylylene dichloride compound include 1,2-xylylene dichloride, 1,3-xylylene dichloride, 1,4-xylylene dichloride, 2-methyl-1,3-xylylene dichloride, 3-methyl-1,4-xylylene dichloride, 2,4-dimethyl-1,3-xylylene dichloride, 2,4,5-trimethyl-1,3-xylylene dichloride, 2,3-dimethyl-1,4-xylylene dichloride, 2,3,5-trimethyl-1,4-xylylene dichloride, 2-ethyl-1,3-xylylene dichloride, 2,4-diethyl-1,3-xylylene dichloride, 2,4,5-triethyl-1,3-xylylene dichloride, 2,3-diethyl-1,4-xylylene dichloride, 2,3,5-triethyl-1,4-xylylene dichloride and the like.

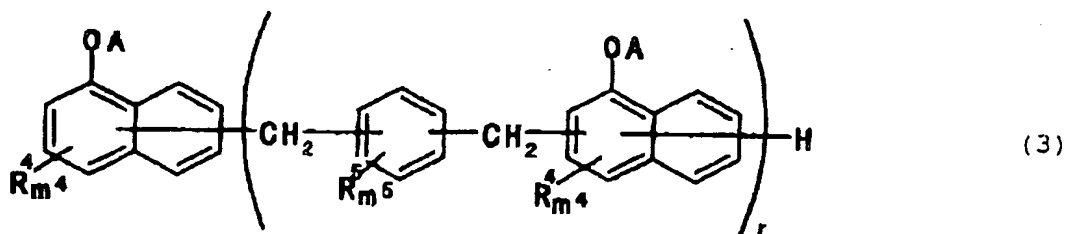
Examples of the xylylene dialkyl ether compound include  $\alpha,\alpha'$ -dimethoxy-p-xylene,  $\alpha,\alpha'$ -diethoxy-p-xylene,  $\alpha,\alpha'$ -dimethoxy-o-xylene,  $\alpha,\alpha'$ -diethoxy-o-xylene,  $\alpha,\alpha'$ -dimethoxy-m-xylene,  $\alpha,\alpha'$ -diethoxy-m-xylene, preferably

$\alpha, \alpha'$ -dimethoxy-p-xylene.

[0084]

Further, otherwise (2-4) is typified by an esterified phenol aralkyl resin expressed by a general formula (3) described below. Although not a particular restriction, a good example is an esterified phenol aralkyl resin such that 10 to 100 mol% of inherent phenolic hydroxyl groups of a naphthol-type phenol aralkyl resin made by being derived from naphthol groups and the xylylene dichloride compound or the xylylene dialkyl ether compound in the presence of a Friedel-Crafts catalyst and subjecting free naphthol to vacuum elimination to 0.01% by weight or less is further subjected to aromatic and/or aliphatic acylation denaturation.

[0085]



[0086]

(wherein  $R^4$  represents a hydrogen atom, a halogen atom, an hydroxyl group, a liner, branched or cyclic alkyl

group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^4$  indicates an integer of from 1 to 3 and  $m^4$ 's may be the same or different;  $R^5$  represents a hydrogen atom or a methyl group;  $m^5$  indicates an integer of from 1 to 3; A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit r is an integer in a range of from 1 to 100; and the repeating unit  $r=1$  represents a bisnaphthol derivative.)

[0087]

Preferable examples of the naphthol include one or two or more of  $\alpha$ -naphthol,  $\beta$ -naphthol, methyl naphthol, dimethyl naphthol, trimethyl naphthol, methylethyl naphthol, ethyl naphthol, diethyl naphthol, triethyl naphthol, methyldiethyl naphthol, n-propyl naphthol, di-n-propyl naphthol, isopropyl naphthol, diisopropyl naphthol, dihydroxy naphthalene, tridihydroxy naphthalene and the like, further preferably naphthol and/or methyl naphthol.

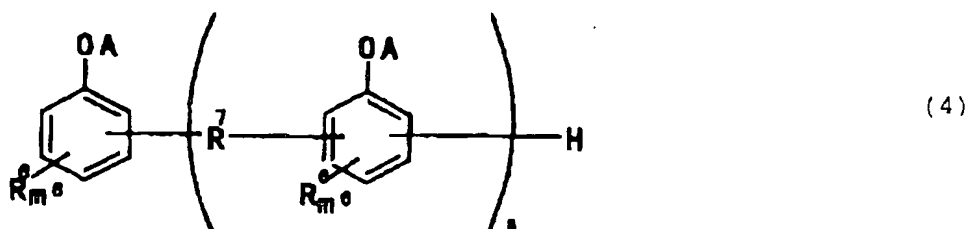
[0088]

(2-5) esterified alicyclic compound modified novolak resin

Representative examples include an esterified alicyclic compound modified novolak resin of the following

formula (4):

[0089]



[0090]

(wherein  $R^6$  represents a hydrogen atom, a halogen atom, an hydroxyl group, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an alkoxy group with carbon number from 1 to 10 or a phenyl group;  $m^6$  indicates an integer of from 1 to 3 and  $m^6$ s may be the same or different;  $R^7$  represents an aliphatic ring of the following formula (6); A represents a hydrogen atom, an aromatic acyl group or an aliphatic acyl group; a molar ratio of the hydrogen atom/the acyl group is in a range of from 100/0 and 0/100; a repeating unit s is an integer in a range of from 1 to 100; and the repeating unit  $s=1$  represents a bisphenol derivative.)

[0091]



[0092]



[0093]

Although not a particular restriction, a preferred example of an esterified alicyclic compound modified novolak resin such that  $R^7$  in the general formula (4) is expressed by the formula (5) is an esterified alicyclic compound modified novolak resin such that 10 to 100 mol% of inherent phenolic hydroxyl groups of a dicyclopentadiene-type modified novolak resin made by being derived from phenol groups and a dicyclopentadiene dichloride compound or a dicyclopentadiene dialkyl ether compound in the presence of a Friedel-Crafts catalyst and subjecting free phenol to vacuum elimination to 0.01% by weight or less is further subjected to aromatic and/or aliphatic acylation denaturation, for example.

[0094]

Preferable examples of the phenol include one or two or more selected from phenol, cresol, xlenol, hydroquinone, methyl hydroquinone, catechol, resorcin, ethyl phenol, diethyl phenol, n-propyl phenol, isopropyl phenol, n-butyl phenol, t-butyl phenol, 2-ethylhexyl phenol, phenyl phenol and the like. Further preferable examples are phenol and/or cresol.

[0095]

Examples of the dicyclopentadiene dialkyl ether compound include dicyclopentadiene dimethyl ether, dicyclopentadiene diethyl ether, dicyclopentadiene dipropyl ether, dicyclopentadiene methylethyl ether and the like.

[0096]

Further, a more preferred specific example of an esterified alicyclic compound modified novolak resin such that  $R^7$  in the general formula (4) is expressed by the formula (6) is an esterified alicyclic compound modified novolak resin such that 10 to 100 mol% of inherent phenolic hydroxyl groups of a dicyclopentadiene-type modified novolak resin made by being derived from the aforementioned phenol groups, with the aforementioned naphthol groups coexist as necessary, and a cyclohexane dichloride compound or a cyclohexane dimethoxy ether compound and/or a cyclohexyl dialkyl ether group typified

by cyclohexane diethyl ether in the presence of a Friedel-Crafts catalyst and subjecting free phenol to vacuum elimination to 0.01% by weight or less is further subjected to aromatic and/or aliphatic acylation modification, for example.

[0097]

(2-6) Polycyclic Aromatic Compound Modified Novolak Resin and/or Esterified composition thereof

A polycyclic aromatic compound modified novolak resin is typified by a condensation polycyclic aromatic compound modified phenol resin that is obtained by causing a tricyclic to tetracyclic condensation polycyclic aromatic hydrocarbon compound derived from light and heavy oil distillate distilled from a high-pressure steam catalytic cracking plant and the like in the petroleum industry, a phenol group, and further, as necessary, formaldehyde to react in the presence of an acid catalyst, and that is made in a manner that the condensation polycyclic aromatic nucleus and a phenol nucleus are bonded by methylene bonding at random in a three-dimensional arrangement, for example. Moreover, an esterified composition thereof is typified by an esterified modified composition such that 10 to 100 mol % of phenolic hydroxyl groups of the condensation polycyclic aromatic phenol resin is further subjected to aromatic

and/or aliphatic acylation denaturation.

[0098]

The tricyclic to tetracyclic condensation polycyclic aromatic hydrocarbon compound derived from light and heavy oil distillate is typified by one having an aromatic carbon rate (an  $f_a$  value) expressed by an aromatic carbon number found by  $^{13}\text{C}$ -NMR in light and heavy oil / a carbon number in light and heavy oil in a range of 0.4 to 0.95, and preferably 0.7 to 0.8, and having an aromatic ring hydrogen rate (an  $H_a$  value) expressed by an aromatic ring hydrogen number found by  $^1\text{H}$ -NMR in light and heavy oil / a hydrogen number in light and heavy oil in a range of 0.2 to 0.8, and preferably 0.35 to 0.6, for example.

[0099]

(2-7) Imidazole-Epoxy Adduct Product and/or Phenol Resin Salt thereof

It is possible to use any adduct product of an imidazole compound having active hydrogen groups and an epoxy resin. For example, a curing accelerator composition that is made by a reaction product of a multifunctional epoxy compound, an imidazole compound, and a phenol novolak resin of less than 2 times the amount of the multifunctional epoxy compound considering the weight thereof as the amount, that has a ratio of epoxy groups in the polyvalent epoxy compound and molecules of the



imidazole compound in a range of (0.8 : 1) to (2.2 : 1), and that exhibits a softening point temperature of 70°C to 150°C can be exemplified. Moreover, an adduct derivative obtained by causing an epoxy resin and an imidazole compound to react and moreover causing a hydroxystyrene resin to react, and further, an adduct derivative of an epoxy resin, a compound having a nitrogen base having no primary amino group in a molecule (including an imidazole compound), and a phenol-formaldehyde resin having a polystyrene conversion weight average molecular weight by GPC of 2000 to 10000 can be exemplified, respectively.

It is preferred to select and use an adduct derivative of an imidazole compound and an epoxy resin having a melting point of 70 to 150°C.

[0100]

(2-8) Micro-encapsulated Imidazole Compound

Representative examples include micro-encapsulated products obtained by encapsulating one of N-cyanoethyl-2-ethyl-4-methylimidazole, 2-ethyl-4-methylimidazole, 2-methylimidazole, 2-n-pentadecylimidazole or the like. Preferable concrete examples include Novacure HX series, as an epoxy resin dispersive compound of the micro-encapsulated products, of Asahi Kasei Epoxy Corporation.

[0101]

(2-9) Methyl Methacrylate Adduct of Alicyclic

#### Diamine

Examples include solid solutions having a softening point temperature of 50°C or more and obtained by reacting 1 mole equivalent of an alicyclic diamine, represented by isophorone diamine, norbornane diamine, with from 1 to 1.9 mole equivalents of a methyl (meta) acrylate ester, represented by methyl acrylate, methyl methacrylate, ethyl acrylate, ethyl methacrylate, and the like.

[0102]

A preferred mode of a curing agent (2) in the two-component-type sealant composition for a plastic liquid crystal cell of the invention is one made by (2-1) alone or (2-2) alone. Moreover, a mode of (2) in the one-component-type sealant composition for a plastic liquid cell of the invention is one made by (2-3) alone, (2-4) alone, (2-5) alone, or a combination of two or more of them. Furthermore, in particular, the most preferable mode of the curing agent (2) is one made by the aromatic ring modified phenol novolak resin (2-4) alone.

[0103]

It is preferred that the curing agent (2) in the invention has an ion conductivity of an aqueous solution obtained by mixing with 10 times by weight of pure water at 40°C to 80°C of 6  $\mu\text{s}/\text{cm}$  or less, more preferably 5  $\mu\text{s}/\text{cm}$  or less, and further preferably 3  $\mu\text{s}/\text{cm}$  or less,

because it is possible to prevent free ion from unnecessarily moving to a liquid crystal phase when a cured product of the sealant composition for a plastic liquid crystal cell of the invention comes in contact with liquid crystal.

Further, the ratio of the curing agent (2) accounting for in the sealant composition for a plastic liquid crystal cell of the invention is 10 to 50% by weight. It is preferred that it is 10% by weight or more, because the curing property of the epoxy resin (1) is favorable and a sealant composition for a plastic liquid crystal cell having high reliability can be substantially obtained, and it is preferred that it is 50% by weight or less, because an unreacted matter of the curing agent can be suppressed from remaining and the crosslinking density of the cured product and the seal adhesion reliability of a liquid crystal cell can be assured to good levels. Concrete examples of the curing agents (2-3) to (2-5), specifically, of esterified compositions thereof will be described in the following, and they may be produced by going through a deionization purifying method in advance so as to have an ion conductivity of an aqueous solution obtained by mixture extraction with 10 times by weight of pure water at 40°C to 80°C of 6  $\mu$ S/cm or less, and the deionization purifying method may be, for example, a free

ion water extraction separation purifying method, a solvent extraction purifying method, a ultrafiltration purifying method and the like, and is not particularly restricted.

[0104]

As the respective esterified compositions of the curing agents (2-3) to (2-5) used in the invention, compounds made by obtaining phenol compounds or phenol resins shown in (2-a) to (2-d) and thereafter esterifying active phenolic hydroxyl groups thereof may be used, and it is possible to preferably use an esterified composition of (2-a) as a concrete example of the esterified composition of (2-3), an esterified compound of (2-b) as a concrete example of the esterified composition of (2-4), an esterified composition of (2-c) as a concrete example of the esterified composition of (2-5), and an esterified composition of (2-d) as a concrete example of the esterified composition of (2-6).

(2-a) phenol novolak resin

(2-b) phenol aralkyl resin

(2-c) alicyclic compound modified novolak resin

(2-d) polycyclic aromatic compound modified novolak resin

A method used for esterifying these phenol resins is a well-known method, and concretely, as described below.

[0105]

In concrete, an esterification agent used for esterifying the phenolic hydroxyl groups as described above may be any of an organic carboxylic acid anhydride, an organic carboxylic acid halide, and an organic carboxylic acid. Depending on the characteristic of an esterification agent by the carbon number of ester to be derived, convenient one can be selected. Specific examples of the esterification agents include acetic anhydride, acetyl chloride, acetyl bromide, acetic acid, propionic anhydride, propionyl chloride, propionyl bromide, propionic acid, butyric anhydride, butyryl chloride, butyric acid, valeraldehyde, valeroyl chloride, valeroyl bromide, valeric acid, pivalic acid, pivaloyl chloride, phenylacetic acid, chloride phenylacetate, 2-phenylpropionic acid, 3-phenylpropionic acid, o-tolyacetic acid, m-tolyacetic acid, p-tolyacetic acid, cumene acid and the like.

[0106]

It is possible to use these esterification agents both independently and by a combination of arbitrary two or more.

The use amount thereof can be 10 mol% or more to hydroxyl groups, and the upper limit is not particularly restricted, so that in a case where esterification is

sufficiently progressed by using in excess, an excess esterification agent can be eliminated after a reaction ends, but realistically, from the point of view of the volume efficiency of a reaction, the cost and so on, it is good to use 10 times by mol or less to hydroxyl groups, preferably 5 times by mol or less, and further preferably 3 times by mol or less.

[0107]

Although concrete reactions vary depending on the kinds of the esterification agents, describing each, a generally used reaction is good for an organic carboxylic acid anhydride.

In specific, by causing an arbitrary amount of organic carboxylic acid anhydride that should be esterified to react with phenolic hydroxyl groups, and thereafter eliminating secondarily produced organic carboxylic acid and excess organic carboxylic acid anhydride by an arbitrary method such as atmospheric distillation, vacuum distillation, washing by water, and washing by water of a weak base like a carbonate, or by a combination thereof, an aimed esterified compound is obtained. That is, when partial esterification is executed, an arbitrary amount to phenolic hydroxyl groups, that is, 10 mol% or more of organic carboxylic acid anhydride is used because an esterified composition such

that 10 mol% or more is esterified is used in the resin composition of the invention, and when complete esterification is executed, an equal mol or more to phenolic hydroxyl groups, considering the economical efficiency and the volume efficiency of a reaction, 10 times by mol% or less is used, though the upper limit is not particularly restricted in the case of doubling as a solvent. Here, this use amount applies to a reaction using an organic carboxylic acid described later.

[0108]

Although reactions of pyridine, piperidine, triethylamine and the like are usually executed in the presence of inactive organic bases in this reaction generally, in a case where the sealant composition for a plastic liquid crystal cell of the invention is used in the electric and electronic field such as a liquid crystal display cell, it must be avoided that nitrogen-containing organic bases thereof remain. Therefore, it is desirable to adopt a process of washing by water finally. However, a reaction sufficiently progresses without use of the organic bases in the esterification reaction. Therefore, it is the most desirable not to use the organic bases in the esterification reaction.

[0109]

It is desirable that a reaction temperature is in a

range of 60°C to 200°C, desirably a range of 80°C to 180°C, and particularly desirably a range of 100°C to 160°C.

Although reaction time is largely influenced by the kind of a reaction substrate and a reaction temperature, it is in a range of 1 to 25 hours approximately, and realistically, it is desirable to determine an ending point while following disappearance of an esterification agent, disappearance of a hydroxyl group and the like by a high-performance liquid chromatography, a gas chromatography and the like.

[0110]

It does not matter if a solvent is used or not in reaction. In a case where a source material having phenolic hydroxyl groups sufficiently melts at a reaction temperature and an esterification agent is liquid, or in a case where it melts or dissolves in a resin at a reaction temperature and there is nothing to hinder a reaction, the reaction can be executed without a solvent.

[0111]

When a solvent is necessary, it is possible to use all solvents that are inactive to a reaction. Examples thereof include aromatic hydrocarbons such as benzene, toluene, xylene, chlorobenzene, o-dichlorobenzene and diphenyl ether, aprotic polar solvents such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-



pyrrolidone, N,N-dimethyl-2-imidazolidinone, dimethyl sulfoxide and sulfolane, ethers such as tetrahydrofuran, dioxane, ethylene glycol dimethyl ether and diethylene glycol dimethyl ether, ketones such as acetone, methyl ethyl ketone and methyl isobutyl ketone, and the like, which can be used individually or by an arbitrary combination.

[0112]

A reaction can be executed at normal pressure, under pressure (in an autoclave) and in vacuum, and the atmosphere of a reaction system can be either in the air or in inactive gas such as nitrogen, argon and helium, but preferably in a nitrogen atmosphere.

[0113]

Next, a reaction in the case of using an organic carboxylic acid halide as an esterification agent will be described. Also in this case, a generally used method can be used.

In specific, an arbitrary amount of organic carboxylic acid halide that should be esterified is made to react with phenolic hydroxyl groups. In this case, it is possible to propose a method of trapping a secondarily produced hydrogen halide by a necessary amount of bases inactive to a reaction of pyridine, piperazine, triethylamine and the like existing in the system, and a

case of discharging outside the system subsequently and speedily during a reaction as gas and trapping by the use of water or alkali trap placed outside the reaction system, but for the reason mentioned before, in order to prevent a nitrogen-containing compound and an ionic compound from mixing in, the method of discharging hydrogen halide gas outside the system speedily during a reaction is preferred.

At this moment, it is more preferable to execute a reaction under air flow of gas inactive to the reaction also.

[0114]

Regarding the use amount of organic carboxylic acid halide, when partial esterification is executed, an arbitrary amount to phenolic hydroxyl groups, that is, 10 mol% or more of an organic carboxylic acid halide can be used because an esterified composition such that 10 mol% or more is esterified is used in the curing agent of the invention, and when complete esterification is executed, equal mol or a small excess amount to phenolic hydroxyl groups can be used, and use of a large excess amount is not particularly restricted, but in consideration of the economical efficiency, the volume efficiency of a reaction and complicatedness of treatment processes after the reaction, it is good to use in a range of 10 times by mol or less, preferably 5 times by mol, and further preferably

3 times by mol to the hydroxyl groups.

[0115]

A reaction temperature, use of a solvent in a reaction and a form of a reaction can be based on those in the aforementioned case of organic carboxylic acid anhydride.

Further, a case of using an organic carboxylic acid as an esterification agent can be based on the case of an organic carboxylic acid anhydride substantially, but an acid catalyst is necessary for a reaction. Examples thereof include mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid and polyphosphoric acid, organic sulfonic acids such as p-toluenesulfonic acid, methanesulfonic acid, ethanesulfonic acid, dimethylsulfonic acid and diethylsulfonic acid, superstrong acids typified by trifluoromethanesulfonic acid, acidic ion exchange resins typified by alkanesulfonic acid type, and superstrong-type ion exchange resins typified by perfluoroalkanesulfonic acid type.

[0116]

The use amount thereof is, to the weight of a raw material: in a range of 0.00001 to 5% by weight, preferably 0.0001 to 1% by weight, and further preferably 0.001 to 0.1% by weight in the case of superstrong acids;

in a range of 1 to 100% by weight, and preferably 10 to 50% by weight in the case of ion exchange resins; and in a range of 0.01 to 10% by weight, and preferably 0.1 to 5% by weight in the other cases. When it is lower than these ranges, a reaction speed decreases, and completion in realistic reaction time is impossible. Moreover, when it is more than these ranges, a secondary reaction cannot be ignored, or an increase of the cost occurs because of complicatedness of a process of eliminating the catalyst and so on.

[0117]

Although the reactions of the three kinds of esterification agents are described above, when it is necessary to obtain an esterified composition having a higher purification degree in each case, it is good to adopt a process of washing by water after a reaction ends. In this case, it is good to wash by the use of a washable solvent such as toluene, xylene, methyl isobutyl ketone, methyl ethyl ketone and ethyl acetate, until acidic components and ionic impurities are not mixed in wastewater of washing any more.

Further, although an esterification ratio thereof is not particularly restricted, and is preferably in a range of 1 mol% to 100 mol%, it is preferably in a range of 50 mol% to 100 mol%, and further preferably 80 mol% to 100

mol%.

[0118]

Further, in the sealant composition for a plastic liquid crystal cell of the invention, a latent epoxy curing agent described below may be used together with the aforementioned curing agent (2) as necessary. Expressing a preferable ratio in the case of using together by the ratio of weights accounting for in the curing agent, the curing agent (2) : the latent epoxy curing agent is in a range of (100 : 1) to (100 : 50).

[0119]

Examples of the latent epoxy curing agent include 4,4-diaminodiphenylsulfone, dicyanodiamide and a derivative thereof, a dibasic acid dihydrazide compound, an aromatic allyl ether compound, boron trifluoride-amine complex, a vinyl ether block carboxylic acid compound and an aromatic allyl ether compound.

[0120]

Although a method for finding the content of the curing resin (2) in the sealant composition for a plastic liquid crystal cell is not particularly restricted, a chromatography fractionation method, an infrared absorption spectrum method, a functional group analysis method, and a solution/solid NMR (nuclear magnetic resonance spectrum) method may be combined properly, for

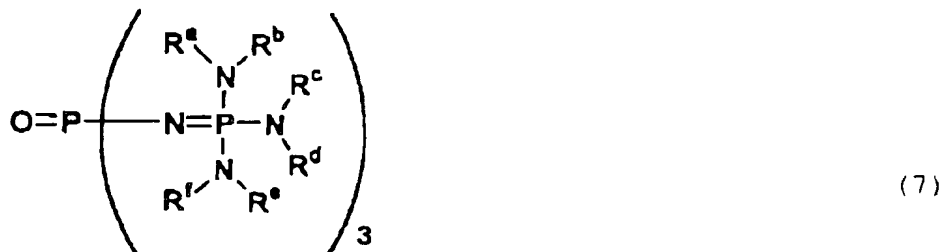
example.

[0121]

[(3) Curing Accelerator]

Examples of the curing accelerator (3) used for the sealant composition for a plastic liquid crystal cell of the invention include a 1,1-dialkyl urea derivative, an imidazole salt, an adduct of a polyamine compound and an epoxy resin, an adduct or a modified derivative thereof of an amine compound and a diisocyanate compound, a trisdimethylaminomethylphenol salt, a 1,8-diazabicyclo(5,4,0)-undecene-7 salt (hereinafter referred to simply as a DBU salt), a 1,5-diazabicyclo(4,3,0)-nonene-5 salt (hereinafter referred to simply as a DBN salt), a 6-dibutylamino-1,8-diazabicyclo(5,4,0)-undecene-7 salt (hereinafter referred to simply as a DADBU salt), a phosphine oxide compound of the following (7) (hereinafter occasionally referred to as phosphazene compound) and the like, and they may be used solely or as a mixture of two or more thereof.

[0122]



[0123]

(wherein from  $R^a$  to  $R^f$  represent a hydrogen atom, a linear, branched or cyclic alkyl group with carbon number from 1 to 10, an aryl group with carbon number from 6 to 10, or an aralkyl group; and Rs may be the same or different.)

Rs of the foregoing formula (7) may be the same or different, and concrete examples thereof include aliphatic hydrocarbons such as a hydrogen atom, a methyl group, ethyl group, a n-propyl group, an isopropyl group, a n-butyl group, a sec-butyl group, a tert-butyl group, a 1-pentyl group, a 2-pentyl group, a 3-pentyl group, a 2-methyl-1-butyl group, an isopentyl group, a tert-pentyl group, a 3-methyl-2-butyl group, a neopentyl group, a n-hexyl group, a 4-methyl-2-pentyl group, a cyclopentyl group, a cyclohexyl group, a 1-heptyl group, a 3-heptyl group, a 1-octyl group, a 2-octyl group, a 2-ethyl-1-hexyl group, an aliphatic hydrocarbon such as a nonyl group or a decyl group, and a aromatic hydrocarbon groups such as a phenyl group, a toluyl group, a benzyl group, and a 1-phenylethyl or 2-phenylethyl group.

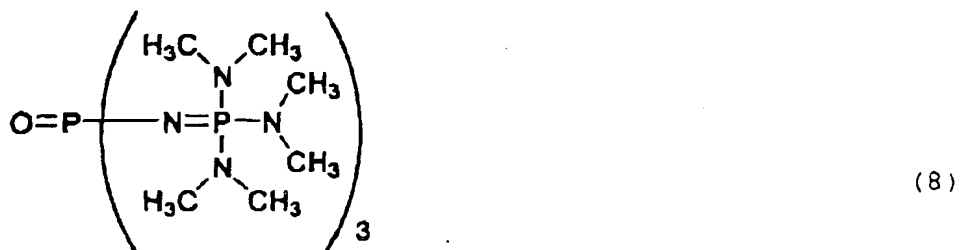
[0124]

Among these, preferable examples include aliphatic hydrocarbon groups with carbon number of from 1 to 6, such as a methyl group, an ethyl group, a n-propyl group, an

isopropyl group, a n-butyl group or a cyclohexyl group, more preferably a methyl group and an ethyl group.

The best state of the phosphine oxide compound is represented by the following general formula (8).

[0125]



[0126]

By the way, the phosphine oxido compound (a phosphazene compound), which is one of phosphine derivatives, as described in G. N. Koian et al. Journal of General Chemistry of The USSR, 55, 1453 (1985), can be synthesized by causing three-molecule iminotrisamino (unsubstituted, monosubstituted, disubstituted) phospholan to react with phosphorus trichloride. Furthermore, when purification is necessary, it is possible to purify by a generally used method such as column chromatography, distillation and recrystallization.

[0127]

The phosphazene compound obtained in this manner is



solid normally.

The use amount of the curing accelerator (3) in the sealant composition for a plastic liquid crystal cell of the invention is 0.01 to 5% by weight, expressing by a ratio accounting for in the whole epoxy resin composition. It is used in a range of 0.1 to 5% by weight preferably, and 1 to 5% by weight more preferably. It is preferred to use 0.01% by weight or more, because the cold cure property of the sealant composition for a plastic liquid crystal cell of the invention can be substantially secured. Moreover, it is preferred to use 5% by weight or less, because it is possible to make the application working property coexistent with the cold cure property substantially.

[0128]

It is preferred to use one having low room-temperature activity and rich storage stability from among the aforementioned, and in this sense, a mode of one selected from among a 1,1-dialkyl urea derivative, DBU salts, DBN salts and a phosphazene compound is good.

Further, it is particularly preferred to select and use the curing accelerator (3) such that the total content of alkali metal obtained by an atomic absorption method of wet solvent is preferably 50 ppm or less, more preferably 30 ppm or less, and particularly preferably 15

ppm or less. Thus, when the cure product of the sealant composition for a plastic liquid crystal cell of the invention comes in contact with liquid crystal, it is possible to prevent free ion from moving to a liquid crystal phase substantially. A purification method for making the total content of alkali metal to be 50 ppm or less is not particularly restricted, and for example, may be an already well-known method such as a solvent extraction purification method.

In the following, the respective examples of the curing accelerators except the phosphazene compound described above will be described in more detail.

[0129]

(1,1-Dialkyl Urea Derivative)

Examples include 3-(p-chlorophenyl)-1,1-dimethyl urea, 3-(o,p-dichlorophenyl)-1,1-dimethyl urea, 2,4-(bis(1,1-dimethyl urea))toluene, 2,6-(bis(1,1-dimethyl urea))toluene and the like.

[0130]

(Imidazole Salt)

Examples include an anhydrous pyromellitic acid salt of 2-ethyl-4-methylimidazole, an anhydrous tetrahydrophthalic acid salt of 2-methylimidazole, an anhydrous tetrahydrophthalic acid salt of 2-ethyl-4-methylimidazole, and the like.

[0131] (Adduct of Polyamine Compound and Epoxy Resin)

Not in order to put particular bounds, but examples include adducts derived from a known polyamine compound and a known epoxy resin, and the like. Specifically, an adduct obtained by reacting an adduct of an epoxy resin and a polyamine with a compound having two or more acidic hydroxyl groups can be exemplified. Examples of the compound having two or more acidic hydroxyl groups include a phenol resin, a modified phenol resin, a polycarboxylic acid and the like.

[0132]

(Adduct or Modified Derivative thereof of Amine Compound and Diisocyanate Compound)

Examples of the adduct of an amine compound and a diisocyanate compound include adducts obtained by reacting a known primary or secondary amine compound with diisocyanate, and the like. Furthermore, examples of the modified derivative of the adduct of an amine compound and a diisocyanate compound include a adduct derivative obtained by reacting under heat N,N-dialkylaminoalkylamine, a cyclic amine and diisocyanate. The examples also include a composition obtained in such a manner that a diisocyanate compound is uniformly made in contact with a surface of particles of the adduct derivative in the form

of powder having a softening point of 60°C or more and containing a tertiary amino group, and the like.

[0133]

(Trisdimethylaminomethylphenol Salt)

Examples of the trisdimethylaminomethylphenol salt include a trisdimethylaminomethylphenol octylic acid salt, a trisdimethylaminomethylphenol oleic acid salt, a trisdimethylaminomethylphenol formate and the like.

[0134]

(DBU Salt)

Examples include a DBU phenol salt, a DBU polyphenol compound salt, a DBU polyphenol salt, a DBU octylic acid salt, a DBU oleic acid salt, a DBU formate and the like.

[0135]

(DBN Salt)

Examples include a DBN phenol salt, a DBN polyphenol compound salt, a DBN polyphenol salt, a DBN octylic acid salt, a DBN oleic acid salt, a DBN formate, a DBN paratoluenesulfonic acid salt and the like.

[0136]

(DADBU Salt)

Examples include a DB phenol salt, a DB polyphenol compound salt, a DB polyphenol salt, a DB octylic acid salt, a DB oleic acid salt, a DB formate, a DB paratoluenesulfonic acid salt and the like.

Not in order to put particular bounds, but for examples of the method for measuring the content proportion of the curing accelerator in the sealant composition for a plastic liquid crystal cell of the invention, a chromatography fractionation method, a water extraction fractionation method, an infrared absorption spectrum method, a phosphorous elemental analysis method, and the like may be performed in combination.

[0137]

[(4) Inorganic Filler]

As the inorganic filler (4) used in the invention, any inorganic filler may be used as far as it can be generally used in the field of electronic materials as an inorganic filler. Specific examples thereof include calcium carbonate, magnesium carbonate, barium sulfate, magnesium sulfate, aluminum silicate, zirconium silicate, iron oxide, titanium oxide, aluminum oxide (alumina), zinc oxide, silicon dioxide, potassium titanate, kaoline, talc, asbestos powder, quartz powder, mica, glass fibers and the like.

It is preferred that a total content of an alkali metal obtained by an atomic absorption spectroscopy of a wet decomposition product is 50 ppm or less, more preferably 30 ppm or less, and further preferably 15 ppm or less. According to the configuration, upon contacting

a cured product of the sealant composition for a plastic liquid crystal cell of the invention with a liquid crystal, migration of free ions to the liquid crystal phase can be practically avoided. Examples of a purification method for obtaining a total content of an alkali metal of 50 ppm or less include an ion exchange purification method and the like without any particular bounds, and the method may be used after forming aqueous solutions in the state of production raw materials.

[0138]

The inorganic filler (4) preferably has a 99% by weight particle diameter on a weight accumulating curve obtained by a laser particle diameter measuring device of a wavelength of 632.8 nm of 5  $\mu\text{m}$  or less, and more preferably a 50% by weight particle diameter on the weight accumulating curve in a range of from 0.005 to 1  $\mu\text{m}$ . Generally, when an inorganic filler having a 99% by weight particle diameter on a weight accumulating curve of 5  $\mu\text{m}$  or less is used, it is preferred since the dimensional stability of the gap width of the liquid crystal panel is further improved.

[0139]

The content ratio of the inorganic filler (4) in the sealant composition for a plastic liquid crystal cell of the invention is from 5 to 50% by weight. It is preferred

that it is contained in 5% by weight or more, in order that workability on screen printing or dispenser coating can be improved. It is also preferred that it is contained in 50% by weight or less, in order that the flowability of the composition can be assured, and thinning upon screen printing and clogging of a dispenser can be suppressed, whereby the coating operation becomes simple. The content ratio of the inorganic filler is preferably from 7 to 45% by weight, and more preferably from 10 to 40% by weight.

[0140]

It is preferred that the inorganic filler (4) is used after subjecting to graft modification with the epoxy resin (1) and the silane coupling agent (5) without any particular bounds. The graft modification may be carried out either a part or the whole of the inorganic filler (4). The graft ratio is expressed by a weight increasing ratio obtained by a repeating solvent washing method, and in general, it is preferred that from 1 to 50 parts by weight of one or both of the epoxy resin (1) and the silane coupling agent (5) is combined chemically on 100 parts by weight of the inorganic filler (4).

[0141]

Examples of the method for obtaining the content ratio of the inorganic filler (4) include a fluorescent X-

ray diffraction spectrum method, an elemental analysis method, a heat decomposition residue method, a method of wet decomposition and atomic absorption analysis and the like without any particular bounds, and they may be performed in combination depending on necessity.

[0142]

[(5) Silane Coupling Agent]

The mixing ratio of the silane coupling agent (5) in the sealant composition for a plastic crystal cell of the invention is from 0.1 to 5% by weight. When it is 0.1% by weight or more, adhesion property to a glass substrate can be assured. Preferably it is 5% by weight or less, in order that the property of suppressing a leak and the adhesion reliability can be assured at the same time. More preferably it is from 0.5 to 3% by weight.

[0143]

Examples of the method for determining the content ratio of the silane coupling agent (5) include a method of heat decomposition gas chromatography, a method of liquid chromatography fractionation and nuclear magnetic resonance spectrum, a method of liquid chromatography and elemental analysis, and the like without any particular bounds.

[0144]

Any of the following examples can be used as the



silane coupling agent (5), and representative examples include a trialkoxysilane compound, a methyldialkoxysilane compound and the like. Preferable examples thereof include  $\gamma$ -glycidoxypropylmethyldimethoxy silane,  $\gamma$ -glycidoxypropyltrimethoxy silane,  $\gamma$ -glycidoxypropylmethyldiethoxy silane,  $\gamma$ -glycidoxypropyltriethoxy silane,  $\gamma$ -aminopropylmethyldimethoxy silane,  $\gamma$ -aminopropyltrimethoxy silane,  $\gamma$ -aminopropylmethyldimethoxy silane,  $\gamma$ -aminopropyltriethoxy silane, N-aminoethyl- $\gamma$ -iminopropylmethyldimethoxy silane, N-aminoethyl- $\gamma$ -aminopropyltrimethoxy silane, N-aminoethyl- $\gamma$ -aminopropyltriethoxy silane, N-phenyl- $\gamma$ -aminopropyltrimethoxy silane, N-phenyl- $\gamma$ -aminopropyltriethoxy silane, N-phenyl- $\gamma$ -aminopropylmethyldimethoxy silane, N-phenyl- $\gamma$ -aminopropylmethyldiethoxy silane,  $\gamma$ -mercaptopropylmethyldimethoxy silane,  $\gamma$ -aminopropyltrimethoxy silane,  $\gamma$ -mercaptopropylmethyldiethoxy silane,  $\gamma$ -mercaptopropyltriethoxy silane,  $\gamma$ -isocyanatepropylmethyldiethoxy silane, isocyanatepropyltriethoxy silane, and the like. Especially, glycidyl silane is the most preferable.

[0145]

[(6) Rubbery Polymer Fine Particles]

In the sealant composition for a plastic liquid crystal cell of the invention, the rubbery polymer fine particles (6) are those having a softening point temperature obtained by a torsional braid analyzer (hereinafter referred to simply as a TBA) called as a torsion pendulum method of 0°C or less, and they preferably contains particles having an average particle diameter of primary particles obtained with an electron microscope of 5  $\mu\text{m}$  or less in an amount of from 1 to 25% by weight (hereinafter occasionally referred to simply as the rubbery polymer fine particles). The average particle diameter of the primary particles of the rubbery polymer fine particles is preferably from 0.01 to 5  $\mu\text{m}$ , more preferably from 0.01 to 3  $\mu\text{m}$ , and further preferably from 0.05 to 2  $\mu\text{m}$ .

[0146]

It is preferred that an amount of 1% by weight or more of the rubbery polymer fine particles is used in the sealant composition for a plastic liquid crystal cell of the invention, in order that the adhesion reliability after a water resistant test at 60 to 80°C of the plastic liquid crystal display cell can be assured. More preferably, the rubbery polymer fine particles are comprised in a range of from 3 to 22.5% by weight, and

further preferably, from 5 to 20% by weight.

[0147]

It is preferred that the softening point temperature of the rubbery polymer fine particles (6) is 0°C or less, in order that the adhesion reliability at low temperatures has a tendency to be further improved. When the primary particle diameter of the rubbery polymer fine particles (6) is 5  $\mu\text{m}$  or less, the gap of the liquid crystal cell can be thin, whereby the using amount of an expensive liquid crystal can be suppressed, and the response speed of the liquid crystal can be improved.

[0148]

Preferable examples of the rubbery polymer fine particles (6) include silicone rubber fine particles having a softening point temperature of -30°C or less and a primary particle diameter in a range of from 0.01 to 3  $\mu\text{m}$ , and/or acrylic rubber fine particles or polyolefin rubber fine particles, and more preferably, the rubbery polymer particles (6) are crosslinked rubbery particles. Preferably, rubbery polymer fine particles (6) have a primary particle diameter of from 0.1 to 1  $\mu\text{m}$  and are crosslinked rubber particles.

[0149]

The following rubbery polymer already known may be selected and used as the rubbery polymer fine particles

(6) as far as the softening point thereof is 0°C or less. Examples thereof include rubbery polymer of acrylic rubber, rubbery polymer of silicone rubber, rubbery polymer of conjugated diene rubber, rubbery polymer of olefin rubber, rubbery polymer of polyester rubber, rubbery polymer of urethane rubber, rubbery polymer having functional groups reacting with a composite rubber and an epoxy group, and the like. Especially, it is preferred that the rubbery polymer have functional group that reacts with an epoxy group.

[0150]

The rubbery polymer fine particles (6) used in the sealant composition for a plastic liquid crystal cell may be used solely or as a mixture of two or more of the foregoing. Specific examples of the rubbery polymer fine particles will be described below.

[0151]

(Rubbery Polymer Fine Particles of Acrylic Rubber)

Examples of the rubbery polymer fine particles of acrylic rubber include fine particles obtained by drying a core/shell emulsion having a core part formed with acrylic rubber, a resin composition obtained by non-aqueous dispersion polymerization of an acrylic monomer in an epoxy resin, a resin composition obtained in such a manner that an acrylic rubber polymer solution obtained by

introducing functional groups that react with an epoxy group is separately prepared and put in or added dropwise in an epoxy resin, followed by mechanical mixing, and then the mixture is subjected to solvent removal or grafting, whereby acrylic rubber fine particles are stably dispersed in the epoxy resin, and the like.

[0152]

(Rubbery Polymer Fine Particles of Silicone Rubber)

Examples of the rubbery polymer fine particles of silicone rubber include a resin composition obtained in such a manner that double bonds are introduced into powder silicone rubber fine particles and an epoxy resin, the double bonds are reacted with a silicone macromonomer having a one-side acrylate group capable of reacting with the double bonds, and then vinyl silicone and hydrogen silicone are charged and subjected to dispersion polymerization, and the like.

[0153]

(Rubbery Polymer Fine Particles of Conjugated Diene Rubber)

As the rubbery polymer fine particles of conjugated diene rubber, known fine particles may be used without any particular bounds, and specific examples thereof include conjugated diene rubbery polymer fine particles obtained by polymerizing or copolymerizing monomers, such as 1,3-butadiene, 1,3-pentadiene, isoprene, 1,3-hexadiene,

chloroprene, and the like. A marketed production may be used as it is. More specific examples of conjugated diene rubber include a copolymer of butadiene and acrylonitrile, a copolymer of butadiene and acrylonitrile having a carboxyl group at an end thereof, a copolymer of butadiene and acrylonitrile having an amino group at an end thereof, and the like.

[0154]

(Rubbery Polymer Fine Particles of Olefin Rubber)

Examples of the rubbery polymer fine particles of olefin rubber include fine particles formed with an amorphous homopolymer of ethylene, propylene, 1-butene, 2-butene, isobutene or the like, or a copolymer or a terpolymer with other monomers capable of being copolymerized, a composition thereof, and the like. It is also possible to use a resin composition obtained in such a manner that after obtaining those commercially available in the form of olefin rubber latex, they are subjected to a dehydration treatment in an epoxy resin, so as to disperse and stabilize the olefin rubber in the epoxy resin.

[0155]

(Rubbery Polymer Fine Particles of Polyester Rubber)

The rubbery polymer fine particles of polyester rubber are fine particles formed with a rubbery polymer

without any particular bounds, in which polyester bonds are contained in the polymer skeleton. Specific examples of the polyester rubber include a low softening point polyester resin derived, in the presence of a polyalcohol compound of a triol or higher depending on necessity, from at least one diol component selected from liquid polysiloxanediol, liquid polyolefindiol, polypropylene glycol, polybutylene glycol and the like, and at least one dibasic acid selected from adipic acid, maleic acid, succinic acid, phthalic acid and the like, a low softening point polyester resin using an acid anhydride instead of the dibasic acid, a low softening point polyester resin derived from a hydroxy polycarboxylic acid, and the like.

[0156]

(Rubbery Polymer Fine Particles of Urethane Rubber)

The rubbery polymer fine particles of urethane rubber are fine particles formed with a rubbery polymer without any particular bounds, in which urethane bonds and/or urea bonds are contained in the rubbery polymer skeleton. Specific examples thereof include rubbery polyurethane obtained by reacting, in the presence of a polyalcohol compound of a triol or higher depending on necessity, a diol component formed with at least one selected from liquid polysiloxanediol, liquid polyolefindiol, polypropylene glycol, polybutylene glycol

and the like, with a diisocyanate compound represented by hexamethylenediisocyanate, isophorone diisocyanate, tolylene diisocyanate, diphenylmethane diisocyanate, norbornane diisocyanate and the like, rubbery polyurethane obtained by reacting, in the presence of a polyalcohol compound of a triol or higher depending on necessity, at least one long-chain diamine component selected from liquid polysiloxanediamine, liquid polyolefindiamine, polypropylene glycol diamine and the like, with a known diisocyanate compound represented by hexamethylenediisocyanate, isophorone diisocyanate, tolylene diisocyanate, diphenylmethane diisocyanate, norbornane diisocyanate and the like, and the like.

[0157]

(Composite Rubber Fine Particles)

Examples of the composite rubber fine particles include fine particles formed with a graft polymer and/or a block polymer, a core/shell polymer, a double layer polymer or the like formed with two or more of the acrylic series, the silicone series, the conjugated diene series, the olefin series, the polyester series and the urethane series described in the foregoing, and the like.

[0158]

(Rubbery Polymer Fine Particles having Functional Groups reacting with Epoxy Group)



Examples of the rubbery polymer fine particles having functional groups reacting with an epoxy group include those obtained by introducing functional groups reacting with an epoxy group into fine particles of the acrylic series, the silicone series, the conjugated diene series, the olefin series, the polyester series or the urethane series described in the foregoing, and the like.

In the rubbery polymer having functional groups reacting with an epoxy group, it is preferred that the weight ratio of the structure derived from a monomer having functional groups reacting with an epoxy group occupied in the rubbery polymer is from 0.1 to 25% by weight.

[0159]

It is preferred that the content of the repeating structure derived from a monomer having functional groups reacting with an epoxy group is from 0.1 to 25% by weight, in order that the adhesion property of the resulting sealant composition for a plastic liquid crystal cell is remarkably improved.

Examples of the functional group reacting with an epoxy group include a mercapto group, an amino group, an imino group, a carboxyl group, an acid anhydride group, an epoxy group, a hydroxyl group and the like.

[0160]

In the rubbery polymer, it is preferred that at least one of the functional groups is introduced in an amount of from 0.01 to 25% by weight, and more preferably from 0.1 to 10% by weight.

Examples of the method for introducing the functional groups include a random copolymerization method of a monomer containing the functional groups and a monomer constituting the main chain polymer, an alternating copolymerization method, a condensation polymerization method, an addition polymerization method, an introducing method by core/shell polymerization, an ion adsorption introduction method, a swelling and impregnating introduction method, a method of graft polymerizing on a polymer forming rubbery particles, and the like without any particular bounds.

Among these, the copolymerization method and the graft polymerization method are preferred since the necessary functional groups can be introduced in the vicinity of the surface of the rubbery polymer fine particles in high efficiency with a small amount.

[0161]

In the sealant composition for a plastic crystal liquid cell of the invention, it is preferred that the rubbery polymer fine particles (6) maintain the form of particles in the epoxy resin.

Examples of the method for confirming that the rubbery polymer particles (6) are present in the form of particles in the epoxy resin include a method, in which a mixture of the epoxy resin (1) having no turbidity and the rubbery polymer fine particles (6) is prepared, and the composition is observed with an optical microscope to confirm the presence of the rubbery polymer particles, a method, in which a micro-cross section of a cured product obtained by adding a necessary amount of a polymericaptan room temperature curing agent or a polyamine room temperature curing agent to the mixture is dyed and sensitized with osmic acid and observed to confirm with a scanning electron microscope (TEM) or a transmission electron microscope (SEM), a method, in which a micro-layer of the cured product is subjected to microscope IR measurement to confirm, and the like without any particular bounds.

[0162]

Examples of the method for confirming that the rubbery polymer fine particles (6) are present in the form of fine particles in the sealant composition for a plastic liquid crystal cell include, without any particular bounds, a method, in which a heat cured product is obtained, a slight cutting surface of the cured product is dyed, and sensitized with osmic acid and observed with a TEM or an

SEM to determine, a method, in which a fracture surface of the cured product obtained in the same manner is subjected to SEM observation, and simultaneously an elemental analysis image is obtained, to be compared with, so as to determine, a method, in which a surface of a cured product is selectively etched in a known method and then observed with a TEM or a SEM to determine, a method, in which the micro-layer of a cured product is measured for a microscope infrared absorption spectrum (microscope IR) to determine, a method, in which the species and the diameter of particles are determined from the components of a gas formed by decomposition upon irradiating the micro-layer of a cured product with a heat ray to determine, and the like.

[0163]

Examples of the method for obtaining the content ratio of the rubbery polymer fine particles (6) contained in the sealant composition for a plastic liquid crystal display cell having been prepared include, without any particular bounds, a method, in which the content ratio of the rubbery polymer fine particles (6) is determined from the analytical curve of the particular absorption spectrum of the rubbery polymer fine particles in an infrared absorption spectrum (IR) of the liquid crystal sealant composition, a method, in which the species of the

rubbery polymer fine particles identified through the IR analysis is obtained, and they are obtained from the attenuation ratio of the modulus ( $G''$ ) in a low temperature region by TBA measurement, which is an indexical amount of the effects that are apparently exerted by the rubbery polymer fine particles, a heat decomposition gas chromatography method, an elemental analysis method, a method, in which the occupied volume of the rubbery polymer fine particles is obtained from plural SEM photographs of a cured product, and they are obtained by specific gravity conversion, a method obtaining from heat decomposition gas analysis, and the like.

In the sealant composition for a plastic liquid crystal cell of the invention, the rubbery polymer fine particles (6) may be previously grafted with the epoxy resin (1) or may not be grafted.

[0164]

[(7) High Softening Point Acrylic Polymer Fine Particles]

As the sealant composition for a plastic liquid crystal cell of the invention, such a sealant composition is further preferred that is formed by adding from 0.1 to 25 parts by weight of high softening point acrylic polymer fine particles (7) described below in 100 parts by weight of a sealant composition for a plastic liquid crystal

display cell containing the foregoing (1) to (6). It is preferable that 0.1 part by weight or more of the high softening point acrylic polymer fine particles (7) is used together, in order to assure a seal adhesion without seal leak and effusion by an primary adhesion process using a vacuum sheet heat press adhesion or rigid sheet heat press adhesion method which is one of the problem to be solved can be obtained. It is preferable that 25% by weight or less of the high softening point acrylic polymer is used together, in order to secure workability on providing gaps enough.

The high softening point acrylic polymer fine particles (7) has a softening point temperature obtained by TBA of 50°C or more and an average particle diameter of primary particles obtained by observing with an electron microscope of 2  $\mu\text{m}$  or less (hereinafter, occasionally referred to simply as the high softening point acrylic polymer fine particles).

[0165]

When the average particle diameter of primary particles of the high softening point acrylic polymer fine particles (7) is 2  $\mu\text{m}$  or less, workability on providing gaps can be obtained. The average particle diameter of primary particles is preferably from 0.01 to 1  $\mu\text{m}$ , and more preferably from 0.2 to 0.5  $\mu\text{m}$ .

The high softening point acrylic polymer particles (7) may be either a crosslinking type or a non-crosslinking type, and the crosslinking type is preferred, with high softening point acrylic polymer fine particles having a micro-crosslinking structure being the most preferred.

[0166]

The high softening point acrylic polymer fine particles having a micro-crosslinking structure can be produced when the amount of a crosslinking monomer is from 0.1 to 50% by weight, preferably from 1 to 10% by weight and most preferably from 1 to 3% by weight, based on the total monomers upon producing the polymer. A gel fraction is one index of the micro-crosslinking degree. This index is obtained in such a manner that 10 g of the high softening point polymer fine particles are dispersed in 50 g of methyl carbitol as a solvent and filtered after stirring at 25°C for one hour, so as to obtain the amount of the filtrate and the content (dissolved amount) of the polymer in the filtrate, which are then applied to the equation  $\text{Gel fraction (\%)} = (\text{dissolved amount}/10 \text{ g}) \times 100$ . The gel fraction index is preferably from 0 to 99%, and more preferably from 80 to 95%.

[0167]

The high softening point acrylic polymer fine

particles preferably have a solubility parameter (SP value) calculated from the chemical structural formula in a range of from 9 to 11, and more preferably in a range of from 9.3 to 10.5.

Examples of the high softening point acrylic polymer fine particles (7) include, for example, a polymer having polymethyl methacrylate as a main component of a micro-crosslinking type obtained by copolymerizing with from 0.1 to 50% by weight of a crosslinking monomer, a polymethyl methacrylate polymer having an ionomer structure in an amount of from 0.1 to 50% by weight, and the like. In the high softening point acrylic polymer fine particles, it is preferred that one functional group such as an epoxy group, an amino group, an imino group, a mercapto group, a carboxyl group and the like is introduced on the surface of the particles.

More preferably, they have a softening point temperature of from 60 to 150°C and a primary particle diameter of from 0.01 to 2  $\mu\text{m}$ .

[0168]

Incidentally, in the sealant composition for a plastic liquid crystal cell of the invention, the rubbery polymer fine particles (6) and the high softening point acrylic polymer fine particles (7) may be previously formed as a composite, and for example, an aspect of a so-



called core/shell composite fine particles A of the rubbery polymer fine particles (6) and the high softening point acrylic polymer fine particles (7), in which the rubbery polymer fine particles (6) form a core phase, and the high softening point acrylic polymer fine particles (7) form a shell phase, is included. An aspect using a core/shell composite fine particles B of the inverted structure, in which the high softening point acrylic polymer fine particles (7) form a core phase, and the rubbery polymer fine particles (6) form a shell phase, is also included. Upon forming the composite, it is preferred to use the former core/shell composite fine particles A.

In the core/shell composite fine particles A having the rubbery polymer fine particles (6) as a core phase, the weight ratio of the core phase and the shell phase is preferably from 0.3 to 2 for the shell phase where the core phase is 1. As an example of the core/shell high softening point polymer fine particles A, "Zeon F-351", a trade name, produced by Nippon Zeon Co., Ltd. is easily available and can be preferably used.

[0169]

Examples of the method for determining the content ratio of the high softening point acrylic polymer fine particles (7) in the sealant composition for a plastic

liquid crystal cell include a heat decomposition gas chromatography method, coagulation filtration method and a solid nuclear magnetic resonance spectrum method without any particular bounds.

[0170]

In the sealant composition for a plastic liquid crystal cell of the invention, the composition using together and including from 1 to 20 parts by weight of the high softening point acrylic polymer fine particles (7) per 100 parts by weight of the sealant composition containing from (1) to (6).

[0171]

[(8) Wax]

In the sealant composition for a plastic liquid crystal cell of the invention, it is preferred to use wax (8) depending on necessity. The using ratio of the wax (8) is preferably from 0.1 to 5 parts by weight per 100 parts by weight of the sealant composition for a plastic liquid crystal cell of the invention.

Examples of the method for obtaining the content ratio of the wax (8) in the sealant composition for a plastic liquid crystal cell are not bounded particularly and include a heat decomposition gas chromatography method, a solid nuclear magnetic resonance spectrum method, a method of hydrocarbon solvent extraction and fractional

quantitative determination, and the like.

[0172]

It is the most preferred that the wax is contained in an amount of from 0.1 to 5 parts by weight per 100 parts by weight of the sealant composition for a plastic liquid crystal cell formed with one of the composition containing from (1) to (6) and the composition containing from (1) to (7). When the mixing amount of the wax is from 0.1 to 5 parts by weight per 100 parts by weight of the sealant composition, the water vapor permeability characteristics at 60°C of the cured product under a high temperature and high humidity environment of 60°C and a relative humidity of 95% or more can be further decreased. According thereto, a plastic liquid crystal display cell having high durability can be produced.

[0173]

As the wax (8), it is not bounded particularly and any type of wax may be used. Examples thereof include animal natural wax, vegetable natural wax, mineral natural wax, petroleum wax, synthetic hydrocarbon wax, modified wax, hydrogenated wax and the like. Among these, wax having a melting point of from 70 to 150°C is preferred, and carnauba wax, microcrystalline wax, Fischer-Tropsch wax and modified Fischer-Tropsch wax are particularly preferred.

[0174]

In the sealant composition for a plastic liquid crystal cell of the invention containing the wax (8), it is preferred that the wax is present as independent primary particles in the state before curing the sealant composition for a plastic liquid crystal cell, and the average particle diameter of the primary particles obtained by observation with an electron microscope or an optical microscope is preferably in a range of from 0.01 to 5  $\mu\text{m}$ , and more preferably in an range of from 0.01 to 3  $\mu\text{m}$ .

More specific examples of the wax (8) will be shown below.

[0175]

(Animal natural wax)

Examples include yellow beeswax, whale wax, shellac wax and the like.

[0176]

(Vegetable natural wax)

Examples include carnauba wax, oricury wax, candelilla wax, wood wax, cane wax and the like.

[0177]

(Mineral natural wax)

Examples include montan wax, ozokerite, ceresin and the like.

[0178]

(Petroleum wax)

Examples include paraffin wax, microcrystalline wax and the like.

[0179]

(Synthetic hydrocarbon wax)

Examples include Fischer-Tropsch wax and a derivative thereof, polyethylene wax and a derivative thereof, polypropylene wax and a derivative thereof, and the like.

[0180]

(Modified wax)

Examples include oxide wax, montan wax, acid-modified wax and the like.

[0181]

(Hydrogenated wax)

Examples include amide wax, such as stearic acid amide wax and the like, polyester wax, opal wax and the like. In particular, the most preferred wax is carnauba wax.

[0182]

[(9) Controlling Agent for providing Gaps]

The controlling agent for providing gaps (9) is such a substance that can arbitrarily and accurately control the gap width of the liquid crystal display cell to a

range of from 3 to 7  $\mu\text{m}$ , and any organic substance and inorganic substance can be used as far as it has such a function.

The controlling agent for providing gaps (9) is preferably contained properly depending on necessity in an amount of from 0.1 to 5 parts by weight, and more preferably from 0.5 to 2.5 parts by weight, per 100 parts by weight of the sealant composition for a plastic liquid crystal cell of the invention.

[0183]

Examples of the method for obtaining the content ratio of the controlling agent for providing gaps (9) in the sealant composition for a plastic liquid crystal cell are not bounded particularly and include an SEM image analysis method, a TEM image analysis method, a classification filtration method, a heat decomposition gas chromatography method, a method of fluorescent X-ray diffraction of heat decomposition residue, an elemental analysis method and the like by the cured product.

[0184]

Examples of the controlling agent for providing gaps (9) include inorganic particles or thermosetting polymer particles that are not modified, dissolved or swelled by the epoxy resin (1) and have a vertical and horizontal symmetry, such as true spheres, soccer ball-like particles,

bar-like fibers and the like.

Examples of the inorganic particles as the controlling agent for providing gaps (9) include true spherical silica particles, true spherical alumina particles, glass short fibers, metallic short fibers, metallic powder and the like. Examples of the controlling agent for providing gaps (9) of the organic substance include thermosetting polystyrene true spherical particles, phenol resin thermosetting particles, benzoguanamine resin thermosetting particles and the like.

[0185]

The inorganic particles are preferred examples since the gap accuracy can be controlled with high accuracy.

[0186]

[Other additives]

A solvent, a leveling agent, a pigment, a dye, a plasticizer and a defoaming agent can be used depending on necessity.

[0187]

[Method of preparing the sealant composition for a plastic liquid crystal cell of the invention]

In the preparation of the one-component sealant composition for a plastic liquid crystal cell of the invention, the epoxy resin (1), the curing agent (2), the curing accelerator (3), the inorganic filler (4), the

silane coupling agent (5) and the rubbery polymer fine particles (6) having a softening point temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu\text{m}$  or less are mixed, and depending on necessity, the high softening point acrylic polymer fine particles (7) having a softening point temperature of 50°C or more and an average particle diameter of primary particles of 2  $\mu\text{m}$  or less, the wax (8), the controlling agent for providing gaps (9), the other additives, such as a very small amount of solvent, the leveling agent, the pigment, the dye, the plasticizer, the defoaming agent and the like may be properly added and mixed without any particular bounds..

[0188]

In the preparation of the two-component sealant composition for a plastic liquid crystal cell of the invention, the epoxy resin (1), the curing accelerator (3), the inorganic filler (4), the silane coupling agent (5) and the rubbery polymer fine particles (6) having a softening point temperature of 0°C or less and an average particle diameter of primary particles of 5  $\mu\text{m}$  or less, and moreover, depending on necessity, the high softening point acrylic polymer fine particles (7) having a softening point temperature of 50°C or more and an average particle diameter of primary particles of 2  $\mu\text{m}$  or less,



the wax (8), the controlling agent for providing gaps (9), other additives, such as small amount of the solvent, the leveling agent, the pigment, the dye, the plasticizer, the defoaming agent and the like may be properly added and prepared by mixing, so as to provide a base resin liquid. Examples of the curing agent liquid include a sole liquid of the curing agent (2), a curing agent liquid prepared by mixing the curing agent (2) and the inorganic filler (4), and a curing agent liquid prepared by mixing the curing agent (2), the curing accelerator (3) and the inorganic filler (4)..

[0189]

Mixing can be conducted using a kneading apparatus such as a double arm mixing apparatus, a roll kneader, a biaxial extruder, a wet media dispersing apparatus and the like. After a vacuum defoaming treatment, finally it may be subjected to be filled and sealed in a glass bottle, a plastic vessel or the like, followed by storage and transportation.

[0190]

[Properties of the sealant composition for a plastic liquid crystal cell]

The viscosity before curing of the sealant composition for a plastic liquid crystal cell is not bounded particularly, but preferably in a range of from 1

to 1,000 Pa·s in terms of viscosity at 25°C by an B type viscometer, more preferably in a range of from 5 to 500 Pa·s, and the most preferably in a range of from 10 to 200 Pa·s. The sealant composition for a plastic liquid crystal cell of the invention may be produced by previously preparing to make the viscosity in the foregoing range with a method, such as heat aging and the like.

[0191]

The thixotropic index expressed by a ratio of 10 rpm viscosity value obtained from the deviation rate of 10 revolutions per minute with the same rotor number as the B type viscometer and 1 rpm viscosity value obtained from the deviation rate of 1 revolution (1 rpm viscosity value/ 10 rpm viscosity value) is not bounded particularly, but preferably in a range of from 1 to 3.

[0192]

[Process for producing the plastic liquid crystal display cell]

A process for producing a plastic liquid crystal display cell of the invention is characterized in that the sealant composition for plastic liquid crystal cell of the invention is obtained by printing or dispenser coating on a component part of an adhering seal of a substrate for a plastic liquid crystal cell and pre-curing the substrate

at a temperature of from 50 to 110°C, positioning the substrate and another substrate without the said process as a pair and a heat pressing treatment at from 60 to 100°C to adhere and fix the pair of substrates with an even thickness in a range of from 3 to 7  $\mu\text{m}$ .

At the time, aspect examples omitting the pre-curing process is preferably contained. In the method for producing as pre-curing condition, the temperature is at preferably from 80 to 100°C, and the period of time for the heat treatment is preferably from 5 to 30 minutes. It is preferred that the higher the temperature is, the shorter the period is. A pre-curing treatment exceeding 110°C can produce a plastic liquid crystal cell, but attention is needed since there is a risk that it is difficult to assure the accuracy of the gap width.

[0193]

Examples of the substrate for the plastic liquid crystal cell are not bounded particularly, but include a plastic substrate for producing the liquid crystal cell formed with polyester, polyallylate, polycarbonate, polyethersulfone and the like preferably as object material. In the substrates, it is righteous that a so-called plastic substrate for constructing a liquid crystal cell wherein the necessary portion is equipped with a transparent electrode represented by indium oxide, an

orientation film represented by polyimide, an inorganic ion shielding film and the like is preferably used.

[0194]

The examples of the method for coating the sealant composition for a plastic liquid crystal cell of the invention on the substrate are not bounded particularly and may include a method for coating by screen printing or dispenser coating and the like. Further, after coating and depending on necessity, the pre-curing process, the sealant composition for a plastic liquid crystal cell and substrate are adhered together by the method of heat press adhesion sealing, however, the heat curing conditions of the treatment are, without any particular bounds, from 50 to 100°C for from 0.5 to 24 hours, and preferably from 70 to 90°C for from 1 to 10 hours.

In the case where the heat press adhesion is carried out by a sheet heat press, it may be produced by two steps or plural steps of the heating step and the aging step, in which the pair of substrates are adhered under the conditions that assure temporary adhesion, preferably conditions of from 100 to 110°C for from 2 to 30 minutes not bounded particularly, and then the pair of substrates are taken out by releasing the pressure, followed by complete hardening and curing in a heating oven at the same temperature.

The sheet heat pressing herein means a heat pressing apparatus of adhering every sets. A sheet heat pressing apparatus capable of applying heat in vacuum is known as a vacuum sheet heat pressing, and a sheet heat pressing method of forcibly adhering with applying heat and pressure through heat plates under atmospheric pressure is referred to as a rigid body sheet heat pressing method. Both the sheet heat pressing methods may be used. A multiplaten heat press may be used in the heat press adhesion process instead of the sheet heat pressing.

[0195]

[Plastic liquid crystal display cell]

The plastic liquid crystal display cell of the invention is obtained by printing or dispenser coating on a component part of an adhering seal of a substrate for a plastic liquid crystal cell and pre-curing the substrate at a temperature of from 50 to 110°C, positioning the substrate and another substrate without the said process as a pair, a heat pressing treatment at from 60 to 100°C to adhere and fix the pair of substrates with an even thickness in a range of from 3 to 7  $\mu\text{m}$ , and charging a liquid crystal material to the inside of the cell and sealing the charging hole with a sealant composition for a two-component liquid crystal or a sealant composition for an ultraviolet cured liquid crystal.

Examples of the two-component liquid crystal sealant composition are not bounded particularly and include a two-component liquid crystal sealant composition formed with an epoxy resin and a polyamide curing agent, a two-component sealant composition for a plastic liquid crystal cell of the invention formed with an epoxy resin and a polythiol curing agent, a two-component liquid crystal sealant composition formed with an epoxy resin and a polyamine curing agent, and the like.

[0196]

Examples of the liquid crystal material are not bounded particularly and preferably include a nematic liquid crystal, a ferroelectric liquid crystal and the like.

Preferred examples of the liquid crystal display cell used in the invention include a TN (twisted nematic) type liquid crystal cell proposed by M. Schadt, W. Helfrich, et al., a STN (super twisted nematic) type liquid crystal cell, a ferroelectric type liquid crystal cell proposed by N.A. Clark and S.T. Lagerwall, a liquid crystal display cell provided with thin film transistors (TFT) for the respective pixels, and the like.

[0197]

[EXAMPLES]

The invention will be described in more detail with

reference to the representative examples below, but it is not limited thereto. All percents and parts in the examples mean percents by weight and parts by weight. The raw materials (abbreviated symbols) used in the examples are as follows.

[0198]

[Test Methods]

(Storage Stability Test)

While assuming the 20°C B type viscosity value when 100 parts of the sealant composition for a plastic liquid crystal cell was put and sealed in a vessel made of polyethylene to be 100, the B type viscosity value after 30 days at -10°C is expressed in terms of the rate of change thereof. Rate of change of less than 10% with good storage stability is shown by the symbol B, rate of change of from 11 to 50% with some problems in storage stability is shown by the symbol C and rate of change exceeding 50% with poor storage stability is shown by the symbol D in the examples.

[0199]

(Coating Workability Test)

The sealant composition for a plastic liquid crystal cell sealed and stored in a vessel made of polyethylene at a freezing point or less was taken out and was allowed to be room temperature 25°C over two hours. While assuming

the 25°C B type viscosity value at that time to be 100, the B type viscosity value after allowing to stand at 25°C for 12 hours is expressed in terms of the rate of change thereof. B rate of change of less than 15% with good coating workability is shown by symbol B, rate of change of from 16 to 50% slightly lacking coating workability is shown by symbol C and rate of change exceeding 50% considerably lacking coating workability is shown by symbol D in the examples.

[0200]

(E Type Viscosity Characteristics at from 50 to 100°C of Composition subjecting to B Stage)

The sealant composition for a plastic liquid crystal cell in the respective examples was coated on a smooth releasing film to a thickness of 50  $\mu\text{m}$ , and 0.6 part of a composition subjecting to B stage obtained by a heat treatment at 75°C for 20 minutes was quickly sampled. The composition subjecting to B stage was increased in temperature from 80°C to 120°C at an even rate of 1°C per two minutes, and a temperature-viscosity curve was obtained with an E type viscometer. The lowest viscosity in a range of from 80 to 120°C (hereinafter sometimes referred to as a bottom viscosity) was read from the temperature-viscosity curve. Case where the bottom viscosity is less than 5 Pa·s is shown by symbol D(-), in



case where the bottom viscosity is from 5 to 500 Pa·s is shown by symbol A and case where the bottom viscosity is from 501 to 5,000 Pa·s is shown by symbol B. Case where the bottom viscosity exceeds 5,000 Pa·s is shown by symbol D(+).

[0201]

(Water Vapor Permeability Characteristics)

The sealant composition for a plastic liquid crystal cell in the respective examples was coated on a smooth releasing film to a thickness of 100  $\mu\text{m}$  and subjected to a heat treatment at 75°C for 20 minutes and further to hardening at 90°C for 5 hours, and the resulting cured film was cut out. The cured film was subjected to a water vapor permeability test according to the water vapor permeability test for a moisture proof packing material (cup method) of the Japanese Industrial Standard (JIS) according to JIS 20208, so as to obtain the amount of water vapor penetrated at 60°C for 24 hours per 100  $\mu\text{m}$  in film thickness (unit:  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ ). As a result, the water vapor permeability characteristics of less than 100  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ , whereby the sealant composition for a plastic liquid crystal cell is particularly excellent in low water vapor permeability is shown by symbol A, the 60°C water vapor permeability characteristics of from 101 to 200  $\text{g}/\text{m}^2 \cdot 24\text{hrs}$ , whereby the sealant composition for a plastic

liquid crystal cell is excellent in low water vapor permeability is shown by symbol B, the 60°C water vapor permeability characteristics exceeding 251 g/m<sup>2</sup>·24hrs, whereby the sealant composition for a plastic liquid crystal cell is lacking in low water vapor permeability is shown by symbol D and the 60°C water vapor permeability characteristics of from 201 to 250 g/m<sup>2</sup>·24hrs is shown by symbol C in the examples.

[0202]

(Heat Deformation Temperature of Cured Product)

The sealant composition for a plastic liquid crystal cell in the respective examples was coated on a smooth releasing film to a thickness of 100 μm and subjected to a heat treatment at 75°C for 20 minutes and further to hardening at 90°C for 5 hours, and the resulting cured film was cut out to a small piece (15 mm square). The cured film as measured for TMA (thermomechanical analysis) under increasing temperature from -30°C to 150°C with a rate of 5°C per minute. The inflection point of the distortion amount was assumed to be the heat deformation temperature (T<sub>g</sub>) of the cured product.

[0203]

(Modulus of Cured Product)

The sealant composition for a plastic liquid crystal cell in the respective examples was coated on a smooth

releasing film to a thickness of 100  $\mu\text{m}$  and subjected to a heat treatment at 75°C for 20 minutes and further to hardening at 90°C for 5 hours, and the resulting cured film was cut out to a size of 3 mm  $\times$  500 mm. The cured film was measured for the storage modulus under the environments of from 20 to 80°C by a Vipron viscoelasticity measuring apparatus.

[0204]

(Water Absorption of Cured Product)

The sealant composition for a plastic liquid crystal cell in the respective examples was coated on a smooth releasing film to a thickness of 100  $\mu\text{m}$  and subjected to a heat treatment at 75°C for 20 minutes and further to hardening at 90°C for 5 hours, and the resulting cured film was cut out to a size of 100 mm square. The cured film was immersed in boiling water for 30 minutes to obtain the increasing amount of weight, and the value was divided by the original weight and multiplied by 100 to provide a water absorption.

That is, it is expressed by the following.

Water absorption (%) = (Increasing amount of weight after immersing in boiling water/Weight before test)  $\times$  100

[0205]

(Free Ion Concentration)

10 parts by weight of the sealant composition for a

plastic liquid crystal cell and 10 times the amount thereof of pure water in the respective examples were stirred and mixed to be extracted at 60°C for 30 minutes to provide an aqueous solution, and the ionic conductivity thereof was measured. Ionic conductivity of extracted water of less than 10  $\mu\text{m}/\text{m}$  is shown by symbol B, ionic conductivity of extracted water of from 10.1 to 19.9  $\mu\text{m}/\text{m}$  is shown by symbol C and ionic conductivity of extracted water exceeding 20  $\mu\text{m}/\text{m}$  is shown by symbol D in the examples, respectively.

[0206]

(Adhesion Seal Test)

The plastic liquid crystal display cell produced through the curing process according to the sheet heat press method under the conditions shown in the respective example was observed with the naked eye through a magnifying lens, so as to measure the presence or absence of disorder of a seal line, and the presence or absence of seal failure points due to occurrence of seal leak.

[0207]

(T Peel Releasing Test)

Plastic substrates made of polyether sulfone (produced by Sumitomo Bakelite Co., Ltd.) cut into a width of 25 mm and a length of 100 mm were adhered with the surfaces in contact with a liquid crystal facing each

other by the sealant composition for a plastic liquid crystal cell with a gap width of 10  $\mu$ , so as to form a test piece, and it was measured for a 20°C T peel strength by an Intesco test apparatus. As a result, breakage observed in transparent electrode layer of the substrate and organic underlayer thereof to provide excellent adhesion property is shown by symbol A, cohesive breakage of sealant composition for plastic liquid crystal cell involved to provide good heat resistant adhesion property is shown by symbol B and breakage associated with interface releasing observed to provide problem in heat resistant adhesion force is shown by symbol D in the examples.

[0208]

(Non-effusion Property of Sealant Composition for Plastic Liquid Crystal Display Cell)

Toward the plastic liquid crystal display cell produced through the curing process of the multiplaten heat press method or the sheet heat press method under the conditions described in the respective examples, and a liquid crystal material RC 4087 (produced by Chisso Corp.) having a threshold voltage of the liquid crystal of 1.38 V and  $\Delta\epsilon$  of the liquid crystal of 12.4 was charged from a inlet for charging a liquid crystal by a vacuum method. The charging inlet was then sealed with Structbond ES-302

(produced by Mitsui Chemicals, Inc.). A deflection plate was attached to the front side, and a deflection plate with a reflection plate was attached to the rear side. Thereafter, a driving circuit and the like were mounted on the unit to produce a liquid crystal panel. The evaluation of the non-effusion property was carried out by determining as to whether or not the liquid crystal display function in the vicinity of the sealant in the liquid crystal panel was normally performed from the initial stage of operation. Liquid crystal display function exerted by the seal to assure non-effusion property is shown by symbol B, liquid crystal display function not normally exerted within 1 mm in the vicinity of the seal to be somewhat lack in non-effusion property is shown by symbol C and liquid crystal display panel exceeding 1.1 mm in the vicinity of the seal to be considerably lack in non-effusion property is shown by symbol D.

[0209]

(Seal Function Durability Test)

Toward the plastic liquid crystal display cell produced through the curing process of under the conditions described in the respective examples, a liquid crystal RC 4087 (produced by Chisso Corp.) was charged from an inlet for charging a liquid crystal. The charging

inlet was then sealed with Structbond ES-302 (produced by Mitsui Chemicals, Inc.) to produce a liquid crystal panel. The liquid crystal panel was allowed to stand under an atmosphere of 65°C and RH95% for 250 hours, 500 hours and 1,000 hours and then taken out. A deflection plate was attached to the front side, and a deflection plate with a reflection plate was attached to the rear side. Thereafter, a driving circuit and the like were mounted on the unit to observe the change of the display function. As the measuring conditions under the measuring environment, it was allowed to stand under an atmosphere of 60°C and RH90%. As a result, no occurrence of display unevenness observed after 500 hours is shown in the symbol A, slight display unevenness observed within 500  $\mu\text{m}$  in distance from the seal after 500 hours is shown by symbol B and display unevenness observed beyond 500  $\mu\text{m}$  in distance from the seal in the fringe of the cell to cause considerable deterioration in display function after 500 hours is shown by symbol D in the examples.

[0210]

[Raw Materials Used]

1. Epoxy Resin (1)

As a bifunctional bisphenol F type epoxy resin, "Epiclon 830S" (an epoxy equivalent : 170), a trade name, produced by Dainippon Ink And Chemicals, Inc., and as a

flexible epoxy resin, "Adeka EP4000S" (an epoxy equivalent :260), a trade name, produced by Asahi Denka Kogyo K.K. as a propylene oxide adduct of bisfunctional bisphenol A type epoxy resin, were prepared. As the polyfunctional novolak epoxy resin, "Epo Tohto YDCN-702", a trade name, produced by Tohto Chemical Co., Ltd., which is 0-cresol novolak epoxy resin having an epoxy equivalent of 203 was used.

As the aliphatic polyfunctional epoxy resin, polyoxypropylene glycol diglycidyl ether having an epoxy equivalent of 310, neopentyl glycol diglycidyl ether having an epoxy equivalent of 150, polyoxytetramethylene glycol diglycidyl ether having an epoxy equivalent of 557 and 1,6-hexanediol diglycidyl ether having an epoxy equivalent of 165 were used. As the monofunctional epoxy resin, t-butylphenol monoglycidyl ether was used.

[0211]

## 2. Curing Agent (2)

'Milex 3L' produced by Mitsui Chemicals, Inc. was selected and used as a phenol novolak resin, a resin obtained by subjecting Novolak PSM-4261 produced by Gun Ei Chemical Industry Co., Inc. to benzoyl-esterification in accordance with a synthesis example 3 was selected and used as an esterified phenol novolak resin, and 'ZAIROKKU XLC-2L' (a phenol aralkyl resin composed in a manner that



a phenol nucleus is bonded with a xylene nucleus via methylene linkage) produced by Mitsui Chemicals, Inc., specifically, one having a p-polystyrene conversion weight average molecular weight by GPC of 2460, having a softening point of 73°C, having 0.01% or less of free phenol, having 0.01% or less by content of chlorine atoms, and having an ionic conductivity of extracted water by 10 times by weight of pure water at 60°C of 3  $\mu$ S/cm was selected and used as a phenol aralkyl resin. Moreover, as a polycyclic aromatic compound modified novolak resin, 'High purity FPI-5136,' a product name of a PPF resin produced by Kashima Oil Co., Ltd., having a polystyrene conversion weight average molecular weight by GPC of 640, having a softening point of 75°C, having 0.01% or less of free phenol, and having an ionic conductivity of extracted water by 10 times by weight of pure water at 60°C of 7  $\mu$ S/cm was prepared.

[0212]

As the tetrafunctional mercapto compound, pentaerythritol tetrakis(3-mercaptopropionate) having an ionic conductivity of extracted water by 10 times by weight of pure water at 60°C of 0.5  $\mu$ S/m was used.

As the modified polymercapto derivative, such a reaction product was used that was derived in such a manner that 1 equivalent in terms of active hydrogen of

pentaerythritol tetrakis(3-mercaptopropionate) having an ionic conductivity of extracted water by 10 times by weight of pure water at 60°C of 0.5  $\mu$ S/m was mixed with 0.3 equivalent in terms of active isocyanate group of norbornane diisocyanate, and the mixture was subjected to reaction in the presence of a very small amount of triethanolamine until absorption inherent to an isocyanate group was not observed in IR analysis (infrared absorption spectrum analysis).

[0213]

Further, as a microcapsule-type latent epoxy curing agent of an imidazole compound, 'Novacure-HX-3121HP,' the name of a product of Asahi kasei Corporation was prepared. Here, the Novacure is a 30% microcapsule agent containing composite that has a bisphenol-type epoxy resin as dispersing medium.

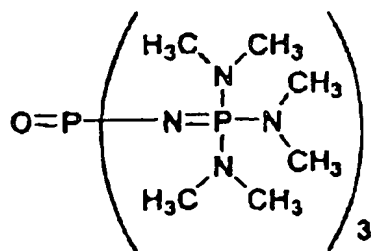
Further, as an imidazole-epoxy adduct-type latent epoxy curing agent, 'Amicure-PN-23' produced by Ajinomoto co., Inc. was used.

[0214]

### 3. Curing Accelerator (3)

Curing accelerator (hereinafter, referred to as PZO) shown in the following general formula (8) by a producing process described in Journal of chemistry of the USSR, 55, p.1453(1985),

[0215]



(8)

[0216]

As the 1,1-dimethyl urea derivative, a dimethylamine adduct of tolylene diisocyanate was prepared, and as the DBU salt, a DBU octylic acid salt and a trisdimethylammonomethylphenol octylic acid salt were prepared. In the case where the curing accelerator was in a solid state, those pulverized by a jet mill to 3  $\mu\text{m}$  or less as the maximum were used depending on necessity.

[0217]

#### 4. Inorganic Filler (4)

As amorphous silica, "MU-120" (average primary particle diameter obtained by observation with electron micrograph: 0.07  $\mu\text{m}$ ), a trade name, produced by Shin-Etsu Chemical Co., Ltd. was used; as spherical silica, barium sulfate having an average particle diameter of 1  $\mu\text{m}$  or less (hereinafter referred to as SO-E1-6) obtained by subjecting "Admafine SO-E1", a trade name, produced by Tatsumori Co., Ltd. to a surface treatment with 6% by weight of  $\gamma$ -glycidoxypolytrimethoxysilane was used; and

as amorphous alumina, high purity alumina powder having an average particle diameter of 0.2  $\mu\text{m}$  (imported) was used.

5. Coupling Agent (5)

$\gamma$ -glycidoxypropyltrimethoxysilane (KBM403, a trade name, produced by Shin-Etsu Chemical Co., Ltd.) was used.

[0218]

6. Rubbery Polymer Fine Particles (6)

One of the compositions prepared by the following Synthesis Example 1 and Synthesis Example 2 was used.

(Synthesis Example 1)

Synthesis of epoxy resin composition (a) containing rubbery polymer fine particles (micro-crosslinking acrylic rubber fine particles, abbreviated as S1)

500 g of Epiclon 830S and 100 g of Adeka EP4000S which is a flexible liquid epoxy resin as a bifunctional epoxy resin, 10 g of methacrylic acid, 1 g of triethanolamine and 50 g of toluene were placed in a 2,000-ml four-neck flask equipped with a stirrer, a gas inlet tube, a thermometer and a condenser tube, and reaction was carried at 110°C for 5 hours under introduction of the air to introduce double bonds. 350 g of butyl acrylate, 20 g of glycidyl methacrylate, 1 g of divinylbenzene, 1 g of azobisdimethylvaleronitrile and 2 g of azobisisobutyronitrile were then added thereto, and reaction was carried out under introduction of nitrogen at

70°C for 3 hours and then at 90°C for 1 hour. Toluene was then removed at 110°C under reduced pressure to obtain an epoxy resin composition (a). The composition (a) was quickly cured at low temperature in the presence of a photocuring catalyst. A fracture surface morphology of the resulting cured product of the epoxy resin composition (a) was observed with an electron microscope to measure the dispersed rubber particle diameter, and thus the micro-crosslinking acrylic rubber fine particles (S1) having an average particle diameter of 0.5  $\mu\text{m}$  were uniformly dispersed. The content of the micro-crosslinking acrylic rubber fine particles (S1) calculated from the amounts of the charged monomer and the remaining monomer was 37.9% by weight.

The softening point temperature of the micro-crosslinking acrylic rubber fine particles (S1) obtained by subjecting the epoxy resin composition (a) to a TBA was -42°C.

[0219]

(Synthesis Example 2)

Synthesis of epoxy resin composition (b) containing rubbery polymer fine particles (micro-crosslinking acrylic rubber fine particles, abbreviated as S2)

500 g of Adeka EP4023S which is a flexible liquid epoxy resin as a bifunctional epoxy resin, 10 g of

methacrylic acid, 1 g of triethanolamine and 50 g of toluene were placed in a 2,000-ml four-neck flask equipped with a stirrer, a gas inlet tube, a thermometer and a condenser tube, and reaction was carried at 110°C for 5 hours under introduction of the air to introduce double bonds. 350 g of 2-ethylhexyl acrylate, 20 g of glycidyl methacrylate, 1 g of 1,6-hexanediol dimethacrylate, 1 g of azobisdimethylvaleronitrile and 2 g of azobisisobutyronitrile were then added thereto, and reaction was carried out under introduction of nitrogen at 70°C for 3 hours and then at 90°C for 1 hour. Toluene was then removed at 110°C under reduced pressure and the composition was quickly cured at low temperature in the presence of a photocuring catalyst. A fracture surface morphology of the resulting cured product of the epoxy resin composition was observed with an electron microscope to measure the dispersed rubber particle diameter, and thus the micro-crosslinking acrylic rubber fine particles (S2) having an average particle diameter of 0.7  $\mu\text{m}$  were uniformly dispersed. The content of the micro-crosslinking acrylic rubber fine particles (S2) calculated from the amounts of the charged monomer and the remaining monomer was 37.9% by weight.

The softening point temperature of the micro-crosslinking acrylic rubber fine particles (S2) obtained

by subjecting the epoxy resin composition (b) to a TBA was -49°C.

[0220]

(Synthesis Example 3)

In a glass container provided with a thermometer, a stirrer, a dropping funnel and a reflux condenser, 107 g of a phenol novolak resin (product name: PSM-4261, hydroxyl group equivalent of 107 g/eq: produced by Gun Ei Chemical Industry Co., Inc.) was charged, and an inside temperature was raised to 125°C. While the inside temperature was kept at 125°C and stirring was executed, 140.6 g of benzoyl chloride was dropped in two hours. After that, a reaction was executed for two hours while the temperature was kept at 125°C, and thereafter, the temperature was further raised to 140°C. After ripening was executed for two hours at temperatures of 140°C to 150°C, evolved hydrochloric acid gas was vacuum distillation on condition of 150°C/10mmHg at the maximum.

[0221]

A resin obtained here was dissolved in 1000 g of toluene, hot water washing by ion exchanged water was executed at temperatures of 60°C to 70°C until waste water became neutral, and thereafter, toluene was distilled on condition of 150°C/5mmHg at the maximum, whereby 210 g of a resin such that hydroxyl groups were completely

benzoylated was obtained. The resin was offered to an example described below as an esterified phenol novolak resin that was 100% esterified.

[0222]

(Example 1)

9.4 parts of t-butylphenol monoglycidyl ether made by separating and drying after washing for 12 hours or more by ion exchange water at 80C° as a liquid epoxy resin, 15.7 parts of neopentyl glycol diglycidyl ether made by separating and drying after washing for 12 hours or more by ion exchange water at 80C°, 31.3 parts of an epoxy resin composition (b) obtained in the synthesis example 2, and 26 parts of a benzoylated phenol novolak resin obtained in the synthesis example 3 as a curing agent were mixed, and the benzylated phenol aralkyl resin was dissolved at 60°C in 30 minutes into the aforementioned epoxy resin.

[0223]

Then, after cooling down to room temperature, further, 1 part of PZO as a curing accelerator, 1.3 parts of amorphous silica 'MU-120,' 12 parts of spherical silica 'SO-E1-6,' and 3.3 parts of KBM403 as a coupling agent were mixed simultaneously, preliminarily mixed by a Dalton mixer, and subsequently kneaded with a three-roll until the solid raw materials became 5  $\mu$ m or less, and the



kneaded product was subjected to a vacuum defoaming treatment to obtain a one-component sealant composition for a plastic liquid crystal cell (P1).

The sealant composition for a plastic liquid crystal cell (P1) is made of a content of the epoxy resin of 44.5%, a content of the rubbery polymer fine particles of 11.9%, a content of the inorganic filler of 13.3%, a content of the silane coupling agent of 3.3%, a content of the curing agent of 26%, and a content of the curing accelerator of 1%.

[0224]

The results of the storage stability test, the results of the coating workability test, the results of the viscosity characteristics subjecting to B stage, the results of the water vapor permeability characteristics, the results of the heat deformation temperature measurement, the results of the free ion concentration measurement, the results of the T peel adhesion test, and the like of the sealant composition for a plastic liquid crystal cell (P1) are shown in Table 1.

[0225]

5 parts of a glass short fiber spacer having a diameter of 5  $\mu\text{m}$  was sufficiently mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (P1) to obtain a composition. The composition was screen

printed on a liquid crystal substrate made of polyether sulfone having a transparent electrode and an orientation film processed (hereinafter referred to as an ITO substrate) to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 80°C for 310 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, fixed by press jig at a pressing pressure of 0.15 MPa/cm<sup>2</sup> at 90°C for 4 hours and were subjected to heat curing and adhesion in the heated oven at 90°C for six hours. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus it was turned out that a desired liquid crystal display cell could be produced.

The results of the observation of the liquid crystal display function of the resulting liquid crystal display cell are also described in Table 1. The results of the seal function durability test carried out by using the resulting liquid crystal display cell are also shown in Table 1.

[0226]

(Example 2)

47.6 parts of a composition obtained by heating and dissolving 26.2 parts of a novolak resin mixed composition (a mixed resin of FPI-5136 and XLC-2L at a ratio of 1 to 1) exhibiting an ion conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 7  $\mu$ S/cm, into 21.4 parts of a mixed resin made by 6.2 parts of 1,6-hexanediol diglycidyl ether exhibiting an ionic conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 0.7  $\mu$ S/cm, 6 parts of polypropylene glycol diglycidyl ether having an epoxy group equivalent of 310 exhibiting an ion conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 3  $\mu$ S/cm, and 9.2 parts of t-butylphenol monoglycidyl ether; 30.5 parts of the epoxy resin composition (b) obtained in the synthesis example 2; 18.3 parts of amorphous alumina having an average particle diameter of 0.2  $\mu$ m; 0.5 part of amorphous silica 'MU-120'; 1.8 parts of Amicure-PN-23; 0.01 part of a DBU octylic acid salt as a curing accelerator; and 1.29 parts of KBM403 as a coupling agent were mixed simultaneously, and preliminarily mixed by a Dalton.

They were then kneaded with three-roll until the solid raw materials became 5  $\mu\text{m}$  or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain the one-component sealant composition for a plastic liquid crystal cell (P2).

[0227]

The sealant composition for a plastic liquid crystal cell (P2) is made of a content of the epoxy resin of 40.3%, a content of the rubbery polymer fine particles of 11.6%, a content of the inorganic filler of 18.8%, a content of the coupling agent of 1.29%, a content of the curing agent made of two kinds of the curing agents of 28%, and a content of the curing accelerator of 0.01%. The results of the storage stability test, the results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, and the results of the T peel adhesion test, the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (P2) are shown in Table 1.

[0228]

5 parts of a glass short fiber having a diameter of 6  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (P2), and they were

sufficiently mixed and defoamed under vacuum to obtain a composition. The composition was charged in a dispenser syringe, dispenser coated on an ITO substrate having a transparent electrode and orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 75°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning and a provisional adhesion process by a rigid sheets heat press at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 90°C for 60 hours. Then, a test of seal adhesion which is the process of curing and adhering in the heated oven at 90 °C for 9 hours was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus it was turned out that a desired liquid crystal display cell could be produced in all of the lot.

The results of the observation of the liquid crystal display function of the resulting cell are also shown in Table 1. The results of the seal function durability test carried out by using the resulting cell are also shown in

Table 1.

[0229]

(Example 3)

30 parts of a composition obtained by heating and dissolving 10 parts of a Milex 3L exhibiting an ion conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 5  $\mu$ S/cm, into 20 parts of a mixed resin made by 5 parts of 1,6-hexanediol diglycidyl ether exhibiting an ionic conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 0.7  $\mu$ S/cm, 5 parts of polyoxytetramethylene glycol diglycidyl exhibiting an ion conductivity of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water at 60°C of 2  $\mu$ S/cm, and 10 parts of t-butylphenol monoglycidyl ether; 20.5 parts of the epoxy resin composition (a) obtained in the synthesis example 1; 38.5 parts of amorphous alumina having an average particle diameter of 0.2  $\mu$ m; 0.49 part of amorphous silica 'MU-120'; 0.01 part of a DBN octylic acid salt as a curing accelerator; and 1 part of KBM403 as a coupling agent were mixed simultaneously, and preliminarily mixed by a Dalton. They were then kneaded with three-roll until the solid raw materials became 5  $\mu$ m or less, and the kneaded product and

10.5 parts of Novacure-HX-3121HP were added into 89.5 parts of the resin composition subjected to a vacuum defoaming treatment so as to obtain the one-component sealant composition for a plastic liquid crystal cell (P2).

[0230]

The sealant composition for a plastic liquid crystal cell (P3) had a content of the epoxy resin of 31.73%, a content of the rubbery polymer fine particles of 7.77%, a content of the inorganic filler of 38.99%, a content of the coupling agent of 1%, a content of the curing agent made of two kinds of the curing agents of 20.5%, and a content of the curing accelerator of 0.01%.

[0231]

The results of the storage stability test, the results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test, and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (P3) are shown in Table 1.

[0232]

5 parts of a glass short fiber having a diameter of 6  $\mu\text{m}$  was mixed with 100 parts of the sealant composition

for a plastic liquid crystal cell (P3), and they were sufficiently mixed and defoamed under vacuum to obtain a composition. The composition was charged in a dispenser syringe, dispenser coated on an ITO substrate having a transparent electrode and orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 75°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and a provisional adhesion process by a rigid sheets heat press at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 90°C for 60 minutes. Then, a test of seal adhesion which was subjected to the process of curing and adhering in the heated oven at 90 °C for 9 hours was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus it was turned out that a desired liquid crystal display cell could be produced in all of the lot.

The results of the observation of the liquid crystal display function of the resulting cell are also shown in



Table 1. The results of the seal function durability test carried out by using the resulting cell are also shown in Table 1.

[0233]

(Example 4)

56 parts of the epoxy resin composition (a), 24 parts of Epiclon 830S, 14.5 parts of fine-particle barium sulfate as an inorganic filler, 3 parts of MU-120 of amorphous silica, 2.4 parts of KBM403, and 0.1 part of a dimethylamine adduct of tolylene diisocyanate were mixed simultaneously, preliminarily mixed by a Dalton, and subsequently kneaded with a three-roll until the solid raw materials became 5  $\mu$ m or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a basis solution (P4 - a basis solution) for a two-component sealant composition for a plastic liquid crystal cell (P4).

[0234]

On the other hand, a composition composed of 76 parts of the pentaerythritol tetrakis (3-mercaptopropionate) as a polymercaptan curing agent and 24 parts of barium sulfate were preliminarily mixed by a Dalton, and subsequently kneaded with a three-roll until the solid raw materials became 5  $\mu$ m or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a curing agent solution (P4 - a curing

agent solution) for a two-component sealant composition for a plastic liquid crystal cell (P4).

[0235]

The sealant composition for a plastic liquid crystal cell (P4) is mixture of 10 parts of the aforementioned (P4 - a basis solution) and 5 parts of (P4 - a curing agent solution). The sealant composition for a plastic liquid crystal cell (P4) was excellent in screen printing working aptitude, because only a minute change was observed regarding a viscosity change when it was left untouched for 24 hours at 23°C.

[0236]

The sealant composition for a plastic liquid crystal cell (P4) is made of a content of the epoxy resin of 39.22%, a content of the rubbery polymer fine particles of 14.15%, a content of the inorganic filler of 19.66%, a content of the coupling agent of 1.6%, a content of the curing agent of 25.3%, and a content of the curing accelerator of 0.07%.

The results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of

the sealant composition for a plastic liquid crystal cell (P4) are shown in Table 1.

[0237]

5 parts of a glass short fiber spacer having a diameter of 5  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (P4), and they were sufficiently mixed to obtain a composition. The composition was screen printed on a ITO substrate having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 90°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and were subjected to heat curing and adhesion by a multiplaten heat press method at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 80°C for 10 hours. Then, a test of seal adhesion was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus a desired liquid crystal display cell could be produced.

The results of the observation of the liquid crystal

display function of the cell are also described in Table 1. The results of the seal function durability test carried out by using the cell are also shown in Table 1.

[0238]

(Example 5)

Per 100 parts of the sealant composition for a plastic liquid crystal cell of example 2, the sealant composition for a plastic liquid crystal cell (P5) which has Tg of 100 and 5 parts of a polymethacrylate fine particles having a diameter of primary particles of 0.35  $\mu\text{m}$  as the high softening point acrylic polymer fine particles is obtained.

The composition (P5) was excellent in screen printing working aptitude, because a minute change was observed regarding a viscosity change when it was left untouched for 24 hours at 23°C. The results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (P5) are shown in Table 1.

[0239]

5 parts of a glass short fiber spacer having a

diameter of 5  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (P5), and they were sufficiently mixed to obtain a composition. The composition was screen printed on a ITO substrate having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of the sealant of about 0.5 mm and a thickness thereof of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 90°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and were subjected to heat curing and adhesion by a multiplaten heat press method at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 80°C for 10 hours. Then, a test of seal adhesion was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus a desired liquid crystal display cell could be produced.

Results of the observation of the liquid crystal display function of the resulting cell are also described in Table 1. The results of the seal function durability test carried out by using the resulting cell are also

shown in Table 1.

[0240]

(Example 6)

A curing agent solution composition composed of 84 parts of a modified polymercapto derivative and 16 parts of barium sulfate was made to be (P6 - a curing agent solution) for a sealant composition for a plastic liquid crystal cell (P6).

Then, the sealant composition for a plastic liquid crystal cell (P6) was obtained by mixing 10 parts of the (P4 - a basis solution) obtained in the example 4 and 10 parts of (P6 - a curing agent solution).

The composition (P6) was excellent in screen printing working aptitude, because a minute change was observed regarding a viscosity change when it was left untouched for 24 hours at 23°C.

[0241]

The sealant composition for a plastic liquid crystal cell (P6) is made of a content of the epoxy resin of 29.4%, a content of the rubbery polymer fine particles of 10.6%, a content of the inorganic filler of 16.75%, a content of the coupling agent of 1.2%, a content of the curing agent of 42%, and a content of the curing accelerator of 0.05%.

[0242]

The results of the coating workability test, the

results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (P6) are shown in Table 1.

[0243]

5 parts of a glass short fiber spacer having a diameter of 5  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (P6), and they were mixed to obtain a composition. The composition was screen printed on a ITO substrate having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 90°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and were subjected to heat curing and adhesion by a multiplaten heat press method at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 80°C for 10 hours. Then, a test of seal

adhesion was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus a desired liquid crystal display cell could be produced.

results of the observation of the liquid crystal display function of the resulting cell are also described in Table 1. The results of the seal function durability test carried out by using the resulting cell are also shown in Table 1.

[0244]

(Example 7)

In the same manner as in the example 1 except replacing 5 parts in 17 parts of an esterified phenol aralkyl resin with 5 parts of ZAIROKKU XLC-2L produced by Mitsui Chemicals Inc., which is a non-esterified phenol aralkyl resin, and additionally blending 2  $\mu$ m or less of carnauba wax powder so that a rate accounting for in a sealant composition became 3%, a sealant composition for a plastic liquid crystal cell (P7) was obtained.

The result of evaluation of physical properties of the sealant composition and the result of a test of making a liquid crystal display cell as well as example 1 are described in Table 1.

[0245]

(Example 8)



After 11 parts of Epo Tohto YDCN 702 was dissolved into 20 parts of Epiclon 830S, 30 parts of the epoxy resin composition (a), 30 parts of SO-E1-6 spherical silica, 2.9 parts of MU-120 of amorphous silica, 4 parts of KBM403 and 0.1 part of a trisdimethylaminomethylphenol octylic acid salt were further mixed simultaneously at room temperatures, preliminarily mixed by a Dalton mixer, and subsequently kneaded with a three-roll until the solid raw materials became 5  $\mu\text{m}$  or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a basis solution (P8 - a basis solution) for a two-component sealant composition for a plastic liquid crystal cell (P8).

[0246]

On the other hand, a composition composed of 66 parts of the pentaerythritol tetrakis (3-mercaptopropionate) as a polymercaptan curing agent, 30 parts of the SO-E1-6 and 4 parts of MU-120 were preliminarily mixed by a Dalton mixer, and subsequently kneaded with a three-roll until the solid raw materials became 5  $\mu\text{m}$  or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a curing agent solution (P8 - a curing agent solution) for a two-component sealant composition for a plastic liquid crystal cell (P8).

[0247]

The sealant composition for a plastic liquid crystal cell (P8) was obtained by mixing 10 parts of the (P4 - a basis solution) obtained and 5 parts of (P8 - a curing agent solution).

The composition (P8) was excellent in screen printing working aptitude, because a minute change was observed regarding a viscosity change when it was left untouched for 24 hours at 23°C.

[0248]

The sealant composition for a plastic liquid crystal display cell (P8) is made of a content of the epoxy resin of 34.35%, a content of the rubbery polymer fine particles of 7.58%, a content of the inorganic filler of 33.3%, a content of the coupling agent of 2.7%, a content of the curing agent of 22%, and a content of the curing accelerator of 0.07%.

[0249]

The results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (P8) are shown in Table 1.

[0250]

5 parts of a glass short fiber spacer having a diameter of 5  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal display cell (P8), and they were sufficiently mixed to obtain a composition. The composition was screen printed on a ITO substrate having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 90°C for 15 minutes, an untreated ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and were subjected to heat curing and adhesion by a multiplaten heat press method at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 80°C for 10 hours. Then, a test of seal adhesion was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus a desired liquid crystal display cell could be produced.

[0251]

results of the observation of the liquid crystal display function of the resulting cell are also described

in Table 1. The results of the seal function durability test carried out by using the resulting cell are also shown in Table 1.

[0252]

(Comparative Example 1)

30 parts of a liquid bisphenol A type epoxy resin having an ionic concentration of 62  $\mu\text{S}/\text{cm}$  of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water and a number average molecular weight of about 390 by GPC and containing hydrolytic chlorine in a high concentration, 23.4 parts of polyethylene glycol diglycidyl ether having an ionic concentration of 37  $\mu\text{S}/\text{cm}$  of an aqueous solution obtained by extraction separation by mixing with 10 times by weight of pure water and an epoxy group equivalent of 260 and containing hydrolytic chlorine in a high concentration, 45 parts of the barium sulfate fine particles as a filling agent, 3 parts of Aerosil 380 which is colloidal silica powder, 2.5 parts of KBM403 and 0.1 part of DBU octylic acid salt were mixed simultaneously were preliminarily mixed in a Dalton. They were then kneaded with three rolls until the solid raw materials became 5  $\mu\text{m}$  or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a base resin liquid (Q1 base resin liquid) for a two-component sealant composition for

a plastic liquid crystal display (Q1).

[0253]

Separately, a composition made of 65 parts of "MR-7B" produced by Mitsui Chemicals, Inc. which is a trifunctional thiol as a polymericaptan curing agent, 30 parts of barium sulfate and 5 parts of Aerosil 200 were preliminarily mixed in a Dalton, they were then kneaded with three-roll until the solid raw materials became 5  $\mu$ m or less, and the kneaded product was subjected to a vacuum defoaming treatment to obtain a curing agent liquid (Q1 curing agent liquid) for the two-component sealant composition for a plastic liquid crystal cell (Q1).

[0254]

The sealant composition for a plastic liquid crystal display cell (Q1) was formed by mixing 10 parts of the Q1 base resin liquid and 5 parts of the (Q1) curing agent liquid.

When the composition (Q1) was allowed to stand at 23°C for 24 hours, only slight fluctuation in viscosity change was observed, and the screen printing workability was excellent.

The sealant composition for a plastic liquid crystal cell (Q1) is made of a content of the epoxy resin of 32.94%, a content of the inorganic filler of 43.67%, a content of the coupling agent of 1.67%, a content of the

curing agent of 21.65%, and a content of the curing accelerator of 0.07%.

[0255]

The results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (Q1) are shown in Table 1.

[0256]

5 parts of a glass short fiber spacer having a diameter of 5  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal cell (Q1), and they were sufficiently mixed and defoamed under vacuum to obtain a composition. The composition was screen printed on an ITO substrate having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ISO substrate with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$ . Thereafter, after subjecting the ITO substrate to a heat treatment by a hot air dryer at 70°C for 15 minutes, an untreated

ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and were subjected to heat curing and adhesion by a multiplaten heat press method at a pressing pressure of 0.03 MPa/cm<sup>2</sup> at 80°C for 10 hours. Then, the test of seal adhesion was repeated ten times. As a result, no sample exhibited seal failure points or disorder of seal lines due to occurrence of seal leak, and thus a desired liquid crystal display cell could be produced.

[0257]

The results of the observation of the liquid crystal display function of the resulting cell are also shown in Table 1. The results of the seal function durability test carried out by using the resulting cell are also shown in Table 1.

[0258]

(Comparative Example 2)

40.3 parts of a composition obtained by heating and dissolving 28 parts of a liquid obtained by mixing 23.5 parts of "YH434" produced by Tohto Chemical Co., Ltd., which is a tetrafunctional epoxy resin, with 4.5 parts of methylcarbitol, 5.8 parts of Epiclon 830S and 29.3 parts of Milex 3L having an ionic conductivity of an aqueous solution obtained by extraction separation by mixing with

10 times by weight of pure water of 5  $\mu$ s/cm in 11 parts of methylcarbitol, 20.5 part of amorphous alumina having an average particle diameter of 0.2  $\mu$ m, 1.19 parts of the amorphous silica "MU-120", 3.2 parts of Amicure PN-23, 0.01 part of DBN octylic acid salt as a curing accelerator and 1 part of KBM403 as a coupling agent were mixed simultaneously and were preliminarily mixed in a Dalton. They were then kneaded with three-roll until the solid raw materials became 5  $\mu$ m or less to obtain a sealant composition for a plastic liquid crystal cell (Q2) of one-component type.

[0259]

The sealant composition for a plastic liquid crystal cell (Q2) is made of a content of the epoxy resin of 29.3%, a content of the inorganic filler of 21.69%, a content of the coupling agent of 1%, a content of the curing agent of 32.5%, a content of the curing accelerator made of two kinds of curing accelerators of 0.01%, and a content of the solvent of 15.5%.

[0260]

The results of the storage stability test, the results of the coating workability test, the results of the water vapor permeability characteristics, the results of the viscosity characteristics subjecting to B stage, the results of the heat deformation temperature



measurement, the results of the T peel adhesion test and the results of the free ion concentration measurement of the sealant composition for a plastic liquid crystal cell (Q2) are shown in Table 1.

[0261]

5 parts of a glass short fiber having a diameter of 6  $\mu\text{m}$  was mixed with 100 parts of the sealant composition for a plastic liquid crystal display cell (Q2), and they were sufficiently mixed and defoamed under vacuum to obtain a composition. The composition was charged in a dispenser syringe and dispenser-coated on an ITO substrate of polyether sulfone having a transparent electrode and an orientation film processed to such a pattern that is formed from four cells of a 1-inch size per one substrate arranged vertically and horizontally so as to obtain the ITO substrate of polyether sulfone with a width of about 0.5 mm and a thickness of about from 20 to 22  $\mu\text{m}$  is obtained. After subjecting the ITO substrate to a heat treatment by a hot air dryer at 60°C for 30 minutes, an ITO substrate, which is the other substrate of the pair of substrates, was placed thereon, followed by positioning, and temporarily adhered by a heat press plate by a pressing pressure of 0.03 MPa/cm<sup>2</sup> at a temperature increased from 70 to 120°C over 10 minutes for 20 minutes in total, and then subjected to a

step of placing it in a heating oven at 120°C for 9 hours, so as to produce a liquid crystal display cell for comparison. As a result, while occurrence of seal leak was observed in from 1 to 5 cells among 10 cells, there are no seal failure points or disorder of seal lines.

However, because the adhesion following property on deformation of the substrate was low when the resulting cells was taken out one-by-one and the modulus of the cured product of the sealant was too high, peeling breaking phenomena at the ITO ground interface were observed when a weak stress of from 10 to 50 g as a peel stress was applied to the direction parallel to the seal line. Owing to the fact that the foregoing phenomena were observed, it was easily expected that serious obstructions occurred upon cutting, washing, injection of a liquid crystal, sealing, assembly processing and the like of the resulting plastic liquid crystal display cells.

[0262] TABLE 1

Subject	Example No.	Example Comparative Example				
		1	2	3	4	5
Sealant composition for plastic liquid crystal display cell		P1	P2	P3	P4	P5
Test result of storage stability test		B	B	B	B	B
Test result of coating workability test		B	B	B	B	B
Test result of seal test						
Presence/absence of disorder of seal line		none	none	none	none	none
Presence/absence of occurrence of through hole		none	none	none	none	none
Suitability for heat press		suitable	suitable	suitable	suitable	suitable
Result of water vapor permeability characteristics		A	A	A	B	A
B type viscosity characteristics at 50 to 100°C subjecting B stage		B	B	B	B	B
Heat deformation temperature of cured product (°C)		54	57	52	34	59
Modulus at room temperature to 80°C of cured product Pa		$9 - 13 \times 10^4$	$1 - 5 \times 10^5$	$1 - 5 \times 10^5$	$3 - 5 \times 10^4$	$2 - 7 \times 10^5$
Water absorption of cured product (%) (method of immersing in boiling water for 30 minutes)		0.3	1.2	0.9	1.0	1.0
Measurement result of free ion		B	B	B	B	B

concentration									
Test result of T peel releasing test		B		B			B		
Test result of non-effusion property								A	B
test of cell		B		B			B		
Test result of seal function durability test									
Lapsed for 250 hours		A		A			A	A	A
Lapsed for 500 hours		A		A			A	B	A
Lapsed for 1,000 hours		A		A			A	C	A

Example			Comparative Example		
6	7	8	1	2	
P6	P7	P8	Q1	Q2	
B	B	B	B	B	
B	B	B	B	B	
none none	none none	none none	none none	none present	
suitable	suitable	suitable	suitable	unsuitable	
B	B	B	C	A	
B	B	B	C	D(-)	
23	59	75	5	125	
$7 - 20 \times 10^3$	$1 - 3 \times 10^5$	$1 - 5 \times 10^5$	$2 - 4 \times 10^3$	$3 - 7 \times 10^6$	
1.1	0.5	0.8	$> 10$	0.7	
B	B	B	D(+)	B	
A	B	B	A	D	
B	B	B	B	not measurable	
A	A	A	C	not measurable	
B	A	A	D		
B	A	A	D		

[0263]

[Effect of the Invention]

The sealant composition for a plastic liquid crystal cell of the invention is the one-component,

- A. is good in storage stability and coating workability,
- B. is high in temporary adhesion property after pre-curing,
- C. a cured product thereof has a low modulus and is rich in film adhesion following property,
- D. the sealant composition is applicable to a multiplaten press method and a sheet press heating adhesion method, and is excellent in non-effusion property, non-seal leak property, linearity of seal lines and accurate controllability of gap width,
- E. the cured product is excellent in low water vapor permeability at 60°C,
- F. the cured product is rich in low water absorbing property,
- G. the electroconductive ions migrating from the composition are suppressed to a low level, and
- H. therefore, the sealant has adhesion durability at high temperatures, and a plastic liquid crystal display cell produced by using the sealant composition for a plastic liquid crystal display cell of the invention has

high display stability for a long period of time under a high temperature and high humidity condition.

[0264]

Especially, as it was apparent from Examples 1 to 8, the liquid crystal cells produced with the sealant compositions for a liquid crystal display cell of the invention had such functions at the same time as an E type viscosity characteristics at 50 to 100°C of a composition subjecting to B stage of the sealant composition for a plastic liquid crystal cell itself of from 5 to 5,000 Pa·s, a water vapor permeability of a cured product of the sealant composition for a plastic liquid crystal cell itself at 80°C of 200 g/m<sup>2</sup>·24hrs or less, a heat deformation temperature of less than 100°C, a modulus of the cured product in a range of from  $0.5 \times 10^4$  to  $1 \times 10^6$  Pa, an ionic conductivity of an aqueous solution obtained by extraction by mixing with 10 times by weight of pure water at 60°C of 10  $\mu$ s /cm or less, and the like. As a result, the seal function durability of the resulting liquid crystal display cells exceeded 1,000 hours.

[0265]

On the other hand, in Comparative Example 1, the free ion conductivity of the sealant composition for a plastic liquid crystal cell was 20  $\mu$ s /cm or more, and the cured product had a considerably high water absorption.

Accordingly, as a result of the seal function durability test of the resulting liquid crystal display cell, obviously stable maintenance of the display function was difficult at the time lapsing for 250 hours, and the service life of the liquid crystal display cell was short.

In Comparative Example 2, because the cured product was as hard as having a modulus of  $2 \times 10^6$  Pa or more, the adhesion following property to the plastic substrate was lacked, and as a result, obviously it was difficult to produce a plastic liquid crystal display cell in a stable manner.

[0266]

Consequently, the plastic liquid crystal display cell produced by using the sealant composition for a plastic liquid crystal cell of the invention is characterized in that display stability for a long period of time under a high temperature and high humidity condition can be assured.

It is concluded that the plastic liquid crystal display cell of the invention can be used as a display of a cell phone or a mobile device used under a high temperature and high humidity condition such as inside of cars.



[DOCUMENT] ABSTRACT

[ABSTRACT]

[MEANS OF SOLUTION]

A sealant composition for a plastic liquid crystal cell comprising: (1) from 15 to 83.89% by weight of an epoxy resin having 1.7 or more in weight average of epoxy groups in one molecule and an ionic conductivity of an aqueous solution obtained by extraction by mixing with 10 times by weight of pure water at from 40 to 80°C of 20  $\mu$ s /cm or less, (2) from 10 to 50% by weight of a curing agent of specific resin, having an ionic conductivity of an aqueous solution obtained by extraction by mixing with 10 times by weight of pure water at from 40 to 80°C of 6  $\mu$ s /cm or less, (3) from 0.01 to 5% by weight of a curing accelerator, (4) from 5 to 50% by weight of an inorganic filler, (5) from 0.1 to 5% by weight of a silane coupling agent, and (6) from 1 to 25% by weight of rubbery polymer fine particles.

[EFFECT]

The sealant composition for a plastic liquid crystal display cell of the invention enables to produce a plastic liquid crystal display cell having a high practical use with secured adhesion following property reliability. As a result, the obtained cell is excellent in adhesion reliability to the cell construction

Application No. 2000-185871

substrate and exertion of the uniform liquid crystal display function as well as long sealing reliability exceeding 1000 hours under a high temperature and high humidity environment of 65°C / saturated humidity 95 %.

[Figure to be published] none